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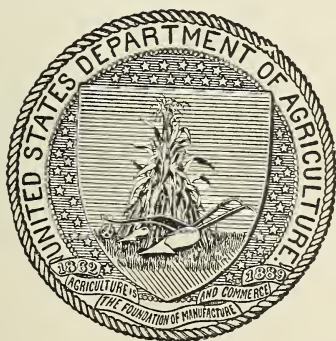# MANUFACTURE OF DENATURED ALCOHOL.

BASED ON THE OPERATIONS OF AN EXPERIMENTAL STILL AT WASHINGTON, D. C., AND A COURSE OF LECTURES DELIVERED IN CONNECTION THEREWITH.

By

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LETTER OF TRANSMITTAL.

Sir: I submit herewith the manuscript reporting the proceedings of the school established by you for teaching the principles of industrial distillation, and also the results obtained by the operation of the experimental industrial distillery erected in harmony with your directions for the purpose of making industrial alcohol, especially from farm and factory wastes. One of the principal objects of this instruction and of these experiments was to teach young men connected with the agricultural colleges and experiment stations the theory and practice of industrial distillation. To this end the distillery was operated in connection with the school of instruction, and various waste materials such as could be found upon the farm, and also the refuse of factories using agricultural products, were employed as raw materials. One of the most useful results of an experimental work of this kind is to ascertain the principal difficulties which stand in the way of a successful establishment of agricultural distillation for industrial purposes, and until these difficulties are thoroughly understood and methods for removing them are established it is not reasonable to expect the development of an agricultural distilling industry in this country. Although now almost three years have elapsed since the passage of the act permitting the use of industrial alcohol free of tax, not a single agricultural distillery has yet been successfully established. It is believed, however, that the information which is contained in the manuscript submitted will be extremely useful in promoting the cause of industrial distillation, especially among agricultural communities. I recommend that it be published as Bulletin No. 130 of the Bureau of Chemistry.

Respectfully,

H. W. Wiley,
Chief of Bureau.

Hon. James Wilson,
Secretary of Agriculture.

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INTRODUCTION.

Since June 7, 1906, a statute of the United States has provided that domestic alcohol may be withdrawn from bond without the payment of an internal-revenue tax, for use in the arts and industries, and for fuel, light, and power, on condition that it shall have been denatured by the admixture of some material which unfits it for use medicinally or as a beverage. The main purpose of this act, known commonly as the "Denatured Alcohol Law," was to reduce the cost of alcohol to the industrial consumer, and it was believed that this end would be attained, not only by the remission of the tax, but in many cases by the special manufacture of industrial alcohol from cheaper materials than have been utilized in the past in making potable alcohol. It became apparent, however, that such materials—like farm waste of different sorts—are most abundant in farming communities remote from the existing centers of the distilling industry and that it would be necessary in securing their utilization to encourage the establishment of small farm distilleries in neighborhoods where such waste is abundantly available. Therefore, in 1907, it was provided, by an amendment to the original act, that special encouragement might be given by the Commissioner of Internal Revenue to distilleries producing industrial alcohol in amounts less than 100 proof gallons daily by the removal of many of the restrictions which are imposed on larger distilleries and on those in which potable alcohol is produced.

It was believed that as a result of this action it would be possible for farmers to make crude alcohol at so low a cost that they could utilize it advantageously upon their farms as a source of power, light, and heat; and it was expected that many communities would establish such agricultural distilleries as were contemplated by the act. Over a year passed, however, without any attempts to make alcohol upon this plan, to the serious disappointment of all who were interested in the matter, and it became apparent that further official encouragement was needed.

By numerous misleading publications a great many people had been led to believe that any waste vegetable material, though of the most worthless character, can profitably be used in making alcohol.

Evidently it was necessary to teach the community which types of material can be so used and which can not. They also had been led to believe that the apparatus of a small distillery can be built very inexpensively and that the manufacturing process is very simple. Evidently it was necessary to furnish accurate information regarding the cost and construction of small plants and regarding the processes of mashing, fermenting, and distilling. They had been led, by cursory examination of the unfamiliar Treasury Regulations, to believe that the latter offered an insuperable barrier to the establishment of small distilleries. Therefore it became essential to provide advisers who should aid in interpreting these regulations.

Having this in mind, the Secretary of Agriculture secured from Congress, in the spring of 1908, the appropriation of a small sum of money for the purpose of demonstrating the manufacture of denatured alcohol "on a scale suitable for utilization by the farmer or associations of farmers." A portion of this appropriation was spent in erecting a small experimental distillery in Washington, in connection with the laboratories of the Bureau of Chemistry; and on completion of the plant invitations were sent to the agricultural experiment stations of the several States, offering to their representatives, and to others who might be interested, full opportunities for instruction in the practice of distilling industrial alcohol. Furthermore, a course of lectures was organized, given in part by employees of the Department and in part by experts engaged in the distilling business, in order to supplement with instruction in the theory of fermenting and distilling, the practical experience gained in the distillery. It was sought thus to train a number of agricultural experts from all sections of the country, who would be able to advise the farmers in their own localities regarding their individual problems and to contribute to our knowledge of these problems by their own investigations. In order, however, to reach a still larger audience and to furnish in printed form some detailed information regarding the construction and operation of small distilleries, such as is continually sought of this Department, it was also determined to issue the present publication, embodying most of the material given in the lectures, together with a description and plans of a small distilling plant (see page 62).

The chapters forming the first section of this report are the work of several chemists of the Bureau named on the title page, Mr. Sawyer having prepared the sections on alcohol, raw materials, and distillation; Mr. Bryan the section on the chemistry of the carbohydrates; and Mr. Berger the sections on yeasting, fermentation, and distillery tests. The lectures given in the latter part of the bulletin are there accredited to the respective authors.

ALCOHOLS AND ALCOHOLOMETRY.

CHEMICAL COMPOSITION OF ALCOHOLS.

To many people the term alcohol signifies a single material, a colorless, mobile liquid, which is characterized by a peculiar "spirituous" odor, a sharp and burning taste, ready inflammability, the property of dissolving gums and resins, and the power of producing intoxication when taken internally. According to the popular conception, there are two varieties of this material, which are distinguished by the prefixes "wood" and "grain," indicating the sources from which the material is obtained, and implying differences in odor, flavor, and intoxicating power due to differences between the impurities derived from the respective sources. In fact, however, the word alcohol is a collective term, applying equally to all substances having a certain typical chemical composition. Wood alcohol and grain alcohol, so-called, are merely the two simplest and commonest bodies of this very large class, and are known respectively to the chemist as methyl and ethyl alcohols. Other members of the class, less abundant, but of considerable technical importance, are obtained as by-products in the manufacture of ethyl alcohol, being known in their crude state as "fusel oils."

Alcohols are known to the chemist as compound bodies, being made up of three of the elementary substances carbon, hydrogen, and oxygen. According to our present conceptions, inconceivably small particles—called "atoms"—of the latter substances are united in definite proportions and in accordance with a definite plan to form a molecule of the compound called alcohol. Many other substances—such, for example, as the sugars and certain acids, which will be discussed farther on, also consist of these same three elements; but the alcohols are distinguished from them in composition by essential differences in the arrangement of the atoms within their molecules, and to a somewhat less extent by differences in the proportions of the three elements. On the other hand, alcohols differ among themselves chiefly in regard to the numbers of atoms composing their molecules, and distinctly less in regard to the details of atom arrangement.

The composition of the two alcohols which are of most interest in the present discussion may be represented as follows:

	Methyl alcohol.	Ethyl alcohol.
Empirical formula..........................	CH_4O	C_2H_6O
Rational formula..........................	CH_3OH	C_2H_5OH

$$\text{Structural formula} \quad\quad H-\underset{\underset{H}{|}}{\overset{\overset{H}{|}}{C}}-O-H \quad\quad H-\underset{\underset{H}{|}}{\overset{\overset{H}{|}}{C}}-\underset{\underset{H}{|}}{\overset{\overset{H}{|}}{C}}-O-H$$

The first set of formulæ show nothing more than the elementary composition of the two alcohols. Thus, with regard to methyl alcohol, it is represented that the molecule contains one atom of carbon, represented as C; four atoms of hydrogen, represented as H_4, and one atom of oxygen, represented as O.

The second set of formulæ, in addition to showing the composition, indicate the bodies to be of the alcohol type in consequence of the linkage of the organic radicles CH_3 and C_2H_5 to the hydroxyl radicle OH.

The third set of formulæ are intended further to show the linkages which are presumed to exist between the individual atoms in each molecule. Formulæ of the second type are generally used to denote the composition of an alcohol, although in some cases it may become necessary to resort to the more complicated structural formulæ. Thus, propyl alcohol, a component of fusel oil having the rational formula C_3H_7OH, exists in two forms which, though of the same molecular composition, are different in certain of their properties. The difference between the two is explained as being due to different arrangements of the carbon and hydrogen atoms within the C_3H_7 groups, as is shown in the following formulæ:

<div style="text-align:center">

Normal propyl alcohol.

$$H-\underset{\underset{H}{|}}{\overset{\overset{H}{|}}{C}}-\underset{\underset{H}{|}}{\overset{\overset{H}{|}}{C}}-\underset{\underset{H}{|}}{\overset{\overset{H}{|}}{C}}-OH$$

or

$$CH_3-CH_2-CH_2-OH$$

Iso-propyl alcohol.

$$H-\underset{\underset{H}{|}}{\overset{\overset{H}{|}}{C}}-H$$
$$H-\underset{|}{\overset{|}{C}}-OH$$
$$H-\underset{\underset{H}{|}}{\overset{\overset{|}{}}{C}}-H$$

or

$$\left.\begin{array}{c}CH_3\\CH_3\end{array}\right\rangle CHOH$$

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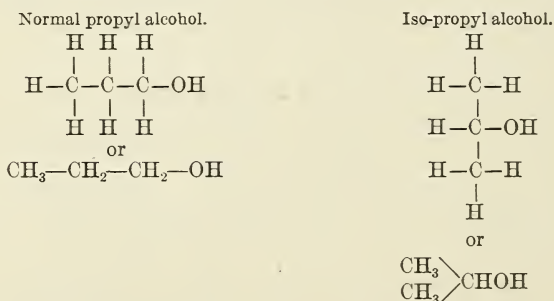
METHYL ALCOHOL.

This substance, the chief constituent of the wood alcohol which is used as a denaturing material, has the formula CH_3OH, and consists by weight of 37.46 per cent of carbon, 12.59 per cent of hydrogen, and 49.95 per cent of oxygen. It is a mobile, colorless liquid, which

when absolutely pure has a pleasant, spirituous odor and a burning taste, slightly different from the odor and taste of ordinary alcohol. Usually, however, it contains impurities which modify its odor considerably; and the grade which is mostly used for denaturing has a very strong and repulsive smell. It boils at 152° F.; and when absolute, or entirely pure and free of water, it has at the standard temperature of 60° F. a specific gravity of 0.79643, referred to water at the same temperature. It mixes freely with water in all proportions, and may be determined in its aqueous solutions by methods which are described in the section on alcoholometry. When concentrated, it burns freely, and is a good solvent for gums and resins; and before the passage of the denatured alcohol law it was used extensively in the preparation of spirit varnishes. It is intoxicating, and is alleged, even in its purest forms, to be more poisonous than ordinary alcohol. It is obtained as a by-product in the manufacture of charcoal by the dry distillation of wood, together with acetone, pyroligneous acid, wood oils, etc., and contains enough of these materials, when crude, to increase its toxicity very greatly. The effects of drinking unpurified wood alcohol are very often fatal; and even if not fatal they commonly involve complete paralysis of the optic nerve, many cases of incurable blindness having been produced in this way. These effects undoubtedly are due to some of the impurities rather than to the alcohol itself.

Specifications for the grade of methyl alcohol which may be used as a denaturant will be found in Regulations No. 30, Revised, United States Internal Revenue, July 15, 1907, pages 23–25.[a]

ETHYL ALCOHOL.

This substance, ordinary commercial alcohol, the production of which is the chief subject of this publication, has the formula C_2H_5OH, and differs from methyl alcohol in that its molecule contains one atom more of carbon and two more of hydrogen. It consists, by weight, of 52.12 per cent of carbon, 13.13 per cent of hydrogen, and 34.75 per cent of oxygen. It is a colorless and mobile liquid, which when pure has a rather faint but characteristic odor, and a burning taste. Unless thoroughly purified, its odor is relatively strong on account of the presence of minute amounts of strong-smelling higher alcohols and other impurities.

It boils at 173° F., and when pure and absolutely free of water it has the specific gravity 0.79387 at the standard temperature, 60° F. Like methyl alcohol, it mixes in all proportions with water, and may

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[a] Copies of this publication may be had free on application to the Commissioner of Internal Revenue, Washington, D. C.

be determined in such mixtures by the methods described in the section on alcoholometry. In a concentrated condition it is a good solvent for gums and resins, and burns freely with a very hot, dim blue flame. As will be explained further on, it is formed in the fermentation of sugar solutions through a decomposition or splitting of the relatively large sugar molecule, and is obtained in a concentrated form by distillation of the fermented liquor which contains it. It is contained to the extent of about 40 or 50 per cent in potable distilled spirits, such as brandy, rum, and whisky, and is responsible for the intoxication produced by these liquors.

HIGHER ALCOHOLS.

The following compounds, together with others which need not be mentioned here, are produced in minute proportions during the alcoholic fermentation, and are sometimes separated from the ethyl alcohol in the form of fusel oil in the course of the distilling process:

Characteristics of some higher alcohols.

Name.	Formula.	Specific gravity.	Boiling point.
			°F.
Normal propyl alcohol	$CH_3-CH_2-CH_2-OH$	0.8044	207.3
Iso-butyl alcohol	$(CH_3)_2-CH-CH_2-OH$	.8020	223.5
Iso-amyl alcohol	$(CH_3)_2-CH-CH_2-CH_2-OH$	.8104	265.5

These compounds differ considerably in physical properties. Thus propyl alcohol resembles ethyl alcohol quite closely in odor, in solubility in water, and in its solvent powers. Butyl and amyl alcohols have odors very different from that of ethyl alcohol, and are thought to be much more poisonous; furthermore, neither will dissolve in water to an unlimited extent. One part of butyl alcohol requires 10.5 parts of water at 64° F., and one part of amyl alcohol requires about 40 parts of water for its solution at the same temperature.

Amyl alcohol occurs in greatest abundance in fusel oil and most analytical methods for fusel-oil determination report it as the sole constituent.

ALDEHYDES, ACIDS, AND ESTERS.

Under certain conditions, the alcohols may be transformed into other bodies of somewhat analogous chemical composition, known, respectively, as aldehydes, acids, and esters. The relationships between bodies of the several types is shown by the following for-

mulæ, which trace the connection between ethyl alcohol and its derivatives, acetaldehyde, acetic acid, and acetic ether:

Alcohol... $CH_3-C{\Large\langle}{\overset{\textstyle H}{\underset{\textstyle OH}{-H}}}$

Acetaldehyde.. $CH_3-C{\Large\langle}{\overset{\textstyle H}{\underset{\textstyle O}{}}}$

Acetic acid.. $CH_3-C{\Large\langle}{\overset{\textstyle OH}{\underset{\textstyle O}{}}}$

Acetic ether... $CH_3-C{\Large\langle}{\overset{\textstyle H_2}{\underset{\textstyle O}{}}}$
$CH_3-C{\Large\langle}{\underset{\textstyle O}{}}$

Alcohol is converted into the corresponding aldehyde, and still further into an acid, by the successive replacement of hydrogen atoms by oxygen. This oxidation will take place to a slight extent when alcohol is freely exposed to the air, the rate of oxidation being immensely increased, under the influence of certain bacteria, in such solutions as the fermented liquors from which the alcohol is distilled. Serious losses may occur if this action is allowed to go on, because alcohol is destroyed in the production of the other bodies, and, furthermore, the acid which may be formed tends to interfere with the progress of the alcoholic fermentation.

A certain proportion of the acetic acid which may be formed always unites with a corresponding amount of unchanged alcohol, to form acetic ether, a body of the ester type. This ester has a certain amount of fuel value, not in excess, however, of that of the alcohol which enters into its composition, and is therefore of no particular value in an industrial alcohol. In potable spirit, on the other hand, this ester and the acid and aldehyde may have much to do with the characteristic flavor and odor of the liquor, to which the particular value of the latter is chiefly due.

ALCOHOLOMETRY.

The true alcoholic strength of any distillate is obtained by the determination of the specific gravity at some standard temperature. Numerous tables for the calculation of the percentage of both methyl and ethyl alcohol have been published, and for various temperatures. The official tables, so far as the alcohol trade in this country is concerned, are those contained in the Gaugers' Manual of the United States Internal Revenue, which give the specific gravity at 60° F. to the fifth decimal place, from that of pure water at 1.00000 to that of absolute ethyl alcohol at 0.79387, at intervals corresponding to one-half volume per cent.

As frequent references are made throughout this publication, as well as in all the literature of the alcohol industry, to weight percentages

and volume percentages, attention is called to the difference and to the need of discrimination between the two. Almost invariably, statements of percentage composition refer to the composition by units of weight. In dealing with alcohol, however, which is always bought and sold by volume, it is usual to state its composition on a volume basis. For example, strong commercial alcohol, such as is referred to in the trade as 95 per cent alcohol, contains in every 100 volumes, at the temperature of 60° F., 95 volumes of absolute alcohol; by weight it contains 92.41 per cent of alcohol and 7.59 per cent of water. A tabulation showing the weight and volume percentages and the specific gravities of aqueous solutions of both methyl and ethyl alcohol, at several concentrations, follows:

Weight and volume percentages of aqueous solutions of methyl and ethyl alcohol of varying concentrations.

Weight.	Methyl alcohol.		Ethyl alcohol.	
	Specific gravity (60° F.).	Volume.	Specific gravity (60° F.).	Volume.
Per cent.		*Per cent.*		*Per cent.*
10	0.98356	12.35	0.98390	12.38
20	.96900	24.33	.97150	24.45
30	.95444	35.94	.95744	36.15
40	.93784	47.09	.93940	47.29
50	.91942	57.71	.91828	57.78
60	.89884	67.70	.89566	67.63
70	.87568	76.95	.87226	76.84
80	.85114	85.47	.84812	85.39
90	.82474	93.17	.82262	93.17
100	.79664	100.00	.79387	100.00

The determination of specific gravity usually is made in specific gravity bottles or by means of a hydrometer. It is desirable to hold as closely as possible to the standard temperature in determining the specific gravity of an alcohol as a preliminary to the estimation of its strength. This is because alcohol and its solutions undergo very considerable alterations in volume and in specific gravity for relatively slight changes in temperature. It is not always convenient, however, in making such observations, to keep the liquid exactly at 60° F., and in such cases it is permissible, after noting its actual temperature at the moment of observation, to apply a correction to the observed specific gravity which will reduce it to the value which would be shown with the solution at 60° F.

It is more expeditious to proceed somewhat differently by having the scale of a spirit hydrometer graduated to indicate the alcoholic strength of a liquid rather than its specific gravity. Instruments made on this plan are prescribed for use by the gaugers of the Internal Revenue Service and are universally employed in the spirit trade in this country. They are graduated, however, in "degrees of proof"

rather than in percentages of alcohol, 1 degree of proof corresponding to one-half of 1 per cent of alcohol by volume. Tables of temperature corrections, together with full instructions for the use of the gaugers' instruments, will be found in the Gaugers' Manual.[a] The instruments themselves may be procured from dealers in chemical apparatus or distillery supplies.

In Great Britain[b] and in the United States the strength of alcoholic liquors is officially stated by reference to an arbitrarily chosen standard of "proof strength." This strength, in the United States, is defined as follows by section 3249 of the Revised Statutes:

Proof spirit shall be held to be that alcoholic liquor which contains one-half its volume of alcohol of a specific gravity of seven thousand nine hundred and thirty-nine ten-thousandths (0.7939) at 60° Fahrenheit.

The same section further authorizes the Commissioner of Internal Revenue to prescribe for use such hydrometers as he may deem necessary. In accordance with this authorization, "the hydrometers furnished to gaugers are so graduated as to indicate the number of parts by volume of proof-spirit equivalent to 100 parts of the liquor at the standard temperature of 60° F. Thus they read 0 for water, 100 for proof spirit, and 200 for absolute alcohol."

Alcohol is bought and sold in small quantities on the basis of its volume, as determined in measures of standard capacity. When handled on a large scale, however, it is more convenient to determine its quantity by weighing. Tables are provided in the Gaugers' Weighing Manual, published by the Treasury Department, which show for spirits of all proofs between 60° and 192° the volumes of alcohol in wine gallons and proof gallons which correspond at 60° F. to weights between about 70 and 400 pounds. Full instructions in the use of the tables are given in the first part of that manual.

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[a] This book and the Gaugers' Weighing Manual, which is mentioned later, may be bought for $1 and for $0.75, respectively, from the Superintendent of Documents, Government Printing Office, Washington, D. C.

[b] British proof spirit is alcohol of such strength that 13 gallons of the spirit have the same weight as 12 gallons of distilled water at 50° F. Its specific gravity at 60° F. is 0.920, and it contains 49.5 weight per cent or 57.3 volume per cent of absolute alcohol.

SOURCES OF ALCOHOL.[a]

CHEMISTRY OF THE CARBOHYDRATES.

Before entering into a consideration of the raw materials that may be used in the manufacture of alcohol, something should be said of the general class of carbohydrates, calling attention to those of special importance in this industry.

From the distiller's point of view, one designates as carbohydrates a group of organic bodies, composed of carbon, hydrogen, and oxygen, which contain six or a multiple of six atoms of carbon, and hydrogen and oxygen in the proportion of water, namely, two atoms of hydrogen and one of oxygen. This definition would not include one class of carbohydrates that occur to some extent in fibrous material, namely, pentosans and pentoses, which contain five atoms of carbon with the hydrogen and oxygen in the form of water.

Embraced in the general term carbohydrates, the alcohol maker would include the various sugars, starch in one form or another, dextrin, cellulose, and certain glucosids. For convenience they will now be considered according to that arrangement.

TRUE SUGARS.

Under this head come first the monosaccharids: (a) *Pentoses*, arabinose and xylose, (b) *hexoses*, dextrose, levulose; second, the *disaccharids*, sucrose and maltose, and third, the *trisaccharid*, raffinose.

MONOSACCHARIDS.

PENTOSES.

Arabinose and xylose.—These compounds, having the formula $C_5H_{10}O_5$, very rarely occur free in nature. They do occur as compounds in the form of xylan and araban (pentosans) $(C_5H_8O_4)_n$ in most woody tissue, or as wood gums or hemicelluloses. In the ordinary products used by the distiller these compounds are present in unappreciable quantities, but in some agricultural wastes that may be used for alcohol making they occur in larger percentages, as in cactus, straw, and wood. On acid hydrolysis, these pentosans pass into the sugars, arabinose and xylose. Both of these reduce Fehling solution, as do the hexose sugars, but are not capable of fermentation, a distinction from the hexoses. Hence their presence might indicate that a higher yield of alcohol should be obtained than is obtained, or that the fermentation has not proceeded far enough. Pentosans and pen-

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toses are recognized by the fact that they yield furfural on distillation of the material with hydrochloric acid. Their separation from the other sugars and from each other is a rather tedious procedure.

<center>HEXOSES.</center>

To this class or division belong the sugars most important to the alcohol maker. Enumerated they are dextrose and levulose. Both have the formula $C_6H_{12}O_6$ and are hence isomeric. Dextrose contains the aldehyde group, while levulose contains the ketone group. If mixed in equal proportions they are termed "invert sugar," if in unequal proportions "reducing sugar," and sometimes wrongly called "glucose," especially in the cane-sugar districts of the South. Both sugars reduce Fehling solution and are capable of fermentation, forming then alcohol and carbon dioxid.

Dextrose.—Dextrose is found widely distributed throughout the vegetable kingdom, as, for instance, in ripe fruits, such as grapes, etc., and in the stems of plants such as sugar cane, sorghum, etc., where it is generally associated with levulose in the form of invert sugar and with sucrose. It is made in large quantities by the acid hydrolysis of starch and dextrin.

Dextrose is soluble in an equal weight of cold water and slightly less of hot water. One part will dissolve in 50 parts of cold and 4.5 parts of boiling absolute alcohol. It crystallizes out of a water solution with one molecule of water, but gradually loses this water at 60° C. It turns the plane of polarized light to the right; when freshly dissolved the specific rotation is $[\alpha]_D = +105$, but it soon drops and becomes constant at about $+52.75$.

The quantitative estimation of dextrose may be made by means of the Fehling solution if it is present alone, but if other reducing sugars are present the separation and estimation are more difficult. Toward acids dextrose is fairly resistant, but on long heating with dilute acids levulinic acid is formed. Alkalies act on this sugar, forming yellow or brown decomposition products. With heat dextrose undergoes decomposition, forming caramel bodies, and is easily and completely fermentable. It yields an osazone with phenyl hydrazin, insoluble in hot water, a distinction from maltose, and has no color reaction with an iodin solution.

Levulose.—This sugar is rightly called fructose, but wrongly termed "fruit sugar." It is found in nature with dextrose in many fruits and in honey. Under ordinary circumstances it is a thick liquid, the crystallized form having been hard to obtain until within the last few years. It can be prepared from invert sugar by precipitation with lime or from inulin by acid hydrolysis. Levulose rotates the plane of polarized light strongly to the left and shows birotation like dextrose. The specific rotation is about $[\alpha]_D = -93.01$. This

is, however, greatly influenced by the concentration of the solution and also the temperature. It reduces Fehling solution like dextrose and also forms an osazone. Its estimation can be accomplished when alone by the Fehling solution or by polarization; in mixtures of sugars, it can be determined by formula from the difference in polarization of the same solution at widely different temperatures. It is easily fermentable, but in a mixture with dextrose the latter ferments first, owing, it is said, to its great diffusion power.

DISACCHARIDS.

Sucrose.—When the layman speaks of sugar sucrose is meant. It is widely found in nature in all parts of plants; in the stem, as in cane and sorghum; in the root, as in beets; in the fruit, as in the sugar palm; and in the sap of trees, principally the maple. The formula $C_{12}H_{22}O_{11}$ is given to this sugar and it comes from the combination of two molecules of a hexose, water being split off, i. e., $C_6H_{12}O_6 + C_6H_{12}O_6 = C_{12}H_{22}O_{11} + H_2O$. Sucrose is very soluble in cold water (one-third part), while in ether and cold absolute alcohol it is insoluble, although boiling absolute alcohol dissolves about 1.25 per cent of its weight. Sucrose rotates the plane of polarized light to the right and does not show birotation. Its specific rotation is variously given, but $[\alpha]_D = +66.5$ may be considered as approximately correct. In itself sucrose is not fermentable nor does it reduce Fehling's solution to any appreciable extent, but by inversion or breaking down of the sucrose into dextrose and levulose it can be made to ferment. This inversion may take place by means of heating with acids or by the action of the invertase of the yeast at a suitable temperature. Sucrose is generally determined by means of the polariscope, either directly or by the formula of Clerget after inversion. In mixtures of other sugars and organic matter, such as in molasses, the reducing power of the solution before and after inversion should be taken. In both cases the reducing power is calculated to invert sugar, one is subtracted from the other, and this difference figured to sucrose by the factor 0.95.

Maltose.—This sugar possesses the same formula as sucrose, namely $C_{12}H_{22}O_{11}$. It is a disaccharid composed of two molecules of dextrose with a molecule of water split off. It does not occur free in nature to any great extent and may be considered as the final product of the action of the enzym diastase on starch. Under the most favorable conditions of temperature for the saccharification of starch, 80 per cent of maltose and 20 per cent of dextrin are produced, but by modifying the conditions as much as 96 per cent of maltose has been obtained.

Maltose is slightly sweet in taste, crystalline in form, and soluble in hot and cold water to the same extent. In absolute alcohol it is insoluble, but in diluted alcohol it is slightly soluble.

Maltose rotates the plane of polarized light strongly to the right (about three times as much as dextrose); it shows birotation. The specific rotation is about $[\alpha]_D = +137.93$. Maltose differs from sucrose in that it reduces the Fehling solution; 100 mg. of dextrose reduce 195 mg. of copper, while 100 mg. of maltose reduce 113 mg. of copper. Maltose can be distinguished from dextrose by its action towards Barfoed's reagent (13.3 grams neutral copper acetate and 5 cc. of 35 per cent acetic acid in 200 cc. of water). Dextrose reduces this solution to cuprous oxid, both in the cold and hot, while maltose does so only by heating. Maltose can be recognized by its osazone (1 part sugar, 2 parts phenyl hydrazin, 2 parts 50 per cent acetic acid, and 20 parts water) being soluble in boiling water and precipitated on cooling. On heating with acids, maltose is broken up into dextrose, but not as easily as sucrose is split into its sugars. The enzym, maltase or glutase, that occurs in the yeast is capable of splitting maltose into dextrose. Diastase has no effect on maltose. Maltose is not fermentable in itself, as was formally held, but has first to be broken down to the hexose dextrose before fermentation can start. This is brought about in actual alcohol making by the enzym of the yeast before mentioned.

TRISACCHARID.

Raffinose.—This sugar is of only passing importance to the distiller. It is found in very small quantities in some beet molasses, but never in cane molasses. It has been found in cotton seed and in a few other seeds and has been claimed to be present in malt. It is a crystalline sugar, soluble in water, but not in methyl alcohol, sweet to the taste, and having a specific rotation of $[\alpha]_D = +104$ to $+105$. On account of its high polarization, its admixture, even in small quantities, in sugar solution would lead to very much higher results for sugar by the polariscope. It does not reduce Fehling's solution, and usually is not fermentable. Both of these attributes are obtained when the sugar is inverted with acids or enzyms. When inverted with acid, it yields one molecule each of levulose, dextrose, and galactose.

Passing from the true sugars, the next group in our classification is starch.

STARCH.

The simple formula for starch is $C_6H_{10}O_5$. The proper expression for starch from a chemical point of view is $(C_6H_{10}O_5) +$ in that it is probable that a number of these simple molecules exist together, variously given at from 5 to 100. Starch turns the plane of polarized light strongly to the right. The specific rotation is variously given, but $[\alpha]_D = +202.0$ might be considered a fair figure.

Starch is hygroscopic, but when treated with strong alcohol readily gives up its water. It is insoluble in alcohol or ether and slightly soluble in water. By heating starch to 160° C. it is changed to the water-soluble variety, then on higher heating without change of weight dextrin is produced. At a still higher temperature decomposition sets in and gaseous substances are formed, which burn, leaving caramel-like bodies. Starch is best recognized by aid of the microscope, though there are some characteristic chemical reactions, notably the one with iodin, in which the starch granules assume a deep-blue to indigo color. Whether this is due to mere solution of iodin in starch or to a formation of a definite compound is a much-mooted question. However, other substances that are capable of forming compounds with iodin rapidly take up the iodin of this blue starch. This reaction is very useful to the alcohol maker. By means of dilute sulphuric and hydrochloric acids, as well as the stronger organic acids—for example, oxalic acid—the starch is changed first into the soluble form, then into dextrin and finally into dextrose. Theoretically, 90 parts of starch will yield 100 parts of dextrose, but in practice it takes nearer 92 to 94 parts. Certain enzyms will do the same work as the acids; as, for example, diastase, which forms maltose as its final product.

Soluble starch.—If ordinary starch is allowed to stand a week in a cold 7.5 per cent solution of hydrochloric acid, stirring or agitating from time to time, and then freeing of acid by washing, a product will result which will go into solution in hot water or even cold and at the same time give all the reactions for the insoluble starch except its solubility in water. The change brought about is claimed to be due to a breaking down of the size of the molecule of starch. Other methods may be employed for preparing soluble starch, and the solubility varies with the method of making and also with the source of the starch. Potato starch, by the treatment described, gives the best soluble starch.

DEXTRIN.

This is an intermediate product between starch and sugar and is formed by the action of enzyms or acids on starch. Formerly it was believed that the dextrin from the acid conversion of starch was different from that from the enzym conversion, but now they seem to be the same in that the dextrin of acid conversion yields maltose before forming dextrose (Brown and Morris). The formula given is $(C_6H_{10}O_5)_n$. Dextrin is a white to yellowish powder with no particular sweet taste, soluble in water, but almost insoluble in alcohol. It does not form an osazone. With iodin the colors range through blue to violet to red and then disappear, depending on the extent of conversion. That giving a blue color is designated as amylodextrin; a red color, erythrodextrin; and colorless, achrodextrin. The violet

color is due to a mixture of amylo and erythro dextrins. The specific rotation varies from the starch end of $+202$ to $+137.04$ on the maltose. The dextrins show some reducing power when treated with Fehling's solution, the amylodextrin the least, increasing through the erythrodextrin and achrodextrin to the maltodextrin, which is the highest. Diastase will reduce dextrin, even it is said that from acid conversion, to maltose. Dextrin is not fermentable until maltodextrin is reached, this form being soluble to a slight extent.

CELLULOSE.

Cellulose is the term applied to a substance which is very widely distributed throughout nature, forming the structural bases of all vegetable organisms, namely, the wall or tissues of the cell. It is never found chemically pure, but contains, or is surrounded by, a series of incrusting combinations called lignins. In young plants or tissues it is purer than in the older ones. In the latter it is heavily incrusted with this material, thereby giving strength. It can be prepared in a pure state by treating wood with various solvents to remove the impurities or incrustants, namely, ether, alcohol, water, alkalies, and acids.

Cellulose is insoluble in the ordinary reagents, but dissolves unchanged in a hydrochloric acid solution of zinc chlorid and also in an ammoniacal cupric oxid solution. Acids react differently with cellulose, depending on the kind of acid, its strength, and the time of contact. Sulphuric acid when diluted has no effect in the cold, but on heating breaks up cellulose into dextrose. The same acid, concentrated, dissolves the cellulose without decomposition when cold, but when heated decomposes it. Hydrochloric acid has about the same effect as sulphuric acid. Nitric acids produce the explosive compounds of nitrocelluloses. Alkalies when rather concentrated produce changes in appearance or structure of the cellulose and when more concentrated produce oxalic acid.

Cellulose and maltose are alike in that by further inversion dextrose is produced. Cellulose reduces Fehling's solution and forms an osazone. It rotates the plane of polarized light to the right, namely, a specific rotation of $[\alpha]_D = +33.70$, and is nonfermentable.

The formula $(C_6H_{10}O_5)_n$ is given to cellulose, being the same as that for starch, gums, dextrin, etc. However, with the same chemical combination of elements, it is seen to possess physical and chemical properties differing from those of the other members.

INULIN, LEVULIN, AND OTHER SUBSTANCES.

These substances occur in some special plants, sometimes in considerable quantities, namely, as inulin in dahlia roots, of which there may be 22 per cent. They have the same formula, $(C_6H_{10}O_5)_n$, as the preceding forms.

Both inulin and levulin on hydrolysis, or treatment with water, yield levulose instead of dextrose. They can be separated as white powders, insoluble in cold water but slightly soluble in hot. They are of only passing value to the distiller, yet may occur in some roots that might be used for alcohol making.

RAW MATERIALS.

Having discussed the carbohydrates which are of chief importance to the distiller, the raw materials from which these substances are obtained will be considered.

These raw materials may be grouped in two main classes, according as their carbohydrate constituent is of a saccharine or a starchy nature; and the materials falling within either group may be subclassed into root crops, stems and leaves, and fruits or seeds. In addition to the natural products there are a few factory wastes, which will be considered in connection with the main group to which they are most nearly allied in composition. Among the miscellaneous lectures will be found several (pages 94–109) relating in detail to various raw materials considered as possible sources of alcohol, though at present some of these, for economic or scientific reasons, can not practicably be put to such use, even though they contain considerable quantities of fermentable material. These papers are submitted, however, because many inquiries concerning the availability of such materials as bananas, cassava, and cactus are received.

The following tabulation shows in a general way the classes of materials available for alcohol making, most of which will be discussed:

Tabulated statement of materials available for alcohol making.

Class.	Saccharine materials.	Starchy materials.
Roots	Sugar beets	Artichoke (Jerusalem). Cassava. Potato. Sweet potato.
Stems and leaves	Agave. Cactus. Sorghum. Stalks of sugar corn. Sugar cane.	
Fruits and seeds	Apple. Banana. Grape. Melon. Orange. Peach. Pear. Pineapple. Prickly pear. Tomato. Watermelon.	Barley. Maize. Oats. Rye.
Trade wastes	Beet molasses. Cane molasses. Corn cannery refuse.	

SACCHARINE MATERIALS.

Sugar beets.—In this country the sugar beet has not as yet been used directly as a source of alcohol. In France, on the contrary, it is used to a very considerable extent, and there is no reason why it may not eventually be profitably utilized for this purpose in this country also. Therefore the following data will be of interest:

The crop of beets varies from 5 to 40 tons per acre, but ordinarily is confined within much narrower limits. Thus the records of several experimental fields cultivated at the agricultural experiment stations in California, Colorado, Michigan, and Wisconsin show yields which run from 8 to 20 tons per acre. It is believed that a yield of 10 tons per acre represents a fair commercial average.

The proportion of sugar in beets may vary between the limits of 10 and 25 per cent; and in the records just referred to it runs from 13 up to 16 per cent; 14 per cent probably represents a fair commercial average.

The data obtained from the results of actual distillery yields in France show that for every 220 pounds of sugar contained in the beets entering the distilleries 15.64 gallons of alcohol of 100 per cent strength were produced, equivalent to 17.4 gallons at 180° proof, which is the minimum strength in this country for alcohol intended for denaturing purposes. On this basis 12.65 pounds of pure cane sugar were consumed for every gallon of alcohol produced at a strength of 180°.

With beets at $4.75 to $5 per ton—the price commonly obtained from the factories in this country—and a sugar content of 14 per cent, the cost of the raw material alone, for a gallon of alcohol at 180°, would be 22.1 cents. This price would be prohibitive under present circumstances.

Agave.—Several varieties of this plant have long been used in Mexico and in the southwestern part of the United States in the preparation of alcoholic beverages, both fermented and distilled. Attempts also have been made within the last few years to utilize this material in the manufacture of industrial alcohol, but so far they have not been attended with success. The problems involved are being studied at the present time in several laboratories.

Cactus.—What is said regarding agave applies also to this plant. The fruit of certain varieties of cactus is discussed in lectures on the cactus and the prickly pear (see pages 105 and 107).

Sorghum.—This is a material which deserves considerable attention in connection with the manufacture of industrial alcohol, on account of the readiness with which it is grown, the extent of the territory in which it flourishes, and the large amount of sugar which certain varieties contain.

Much experimental work has been done by this Department since 1878 with the view of developing the manufacture of sugar from sorghum. Success in this direction has been retarded by the fact that the large proportion of gums and slimy bodies contained in sorghum juice interferes with the crystallization of the sugar to a very serious extent. These gums, however, do not interfere with the utilization of the juice through fermentation. The crop of sorghum is variously estimated at from 5 to 40 tons per acre, but on the basis of field experiments conducted by this Department it is believed usually to be between 10 and 16 tons per acre of whole cane, equivalent to from 7 to 11.5 tons of clean cane. In order to obtain from the cane a material which can be fermented in the distillery it is necessary either to express the juice by heavy pressure in roller mills or to extract it by the diffusion process, which is familiar from its use in beet-sugar factories. The stalk of the plant is much softer than that of sugar cane, so that the juice may be expressed with a lighter mill than is usually used in a cane-sugar factory. A horse-driven mill will easily express 60 per cent of the juice, and with improved mills, such as are used in cane-sugar manufacture, the extraction will exceed that of cane. It is fair to count upon an average extraction of juice corresponding to 65 per cent of the weight of the topped and cleaned sorghum stalks. The sugar content of sorghum juices is shown in the following tabulation, representing results which are embodied in an earlier publication[a] of this Department.

Composition of sorghum juices.

Variety.	Brix.	Sucrose.	Reducing sugars.	Total sugars calculated as dextrose.
		Per cent.	*Per cent.*	*Per cent.*
Black African	19.55	11.69	2.89	15.20
Black African	17.21	11.72	·1.53	13.87
Colman	19.41	14.42	1.10	16.28
Variety 160	17.62	11.55	1.28	13.44
Sink's Hybrid	19.08	13.82	.76	15.31
Undendebule	20.82	15.02	1.29	17.10
Mixed	18.40	12.80	.51	13.98
McLean	20.00	14.18	.59	15.52
No. 161	17.20	11.29	.63	12.52
India and Orange	18.35	12.78	.87	14.32
Mixed	19.64	13.57	.66	15.00
Ubehlana	16.00	10.01	1.30	11.84
Total	18.61	12.74	1.12	13.86

If it is assumed that the sugars of sorghum can be fermented as readily as the sugar of the beet referred to in the preceding section, a yield of 1 gallon of alcohol at 180° proof may be expected from every 13.3 pounds of sugar. With a juice expression of 65 per cent and a

[a] U. S. Dept. Agr., Division of Chemistry, Bul. 34, Record of Experiments with Sorghum in 1891, pp. 23 et seq.

sugar content in the juice corresponding to 13.86 per cent of dextrose, a yield of 13.4 gallons of alcohol per ton of cleaned stalks would be obtained. On this basis, cleaned sorghum stalks delivered at a distillery at $3 per ton would furnish the raw material for a gallon of 180° alcohol at an approximate cost of 22.4 cents.

Stalks of the sugar corn and cannery wastes.—The stalks of the sugar corn contain considerable amounts of sucrose and of reducing sugars, making the material valuable as a source of alcohol. The following analyses, made in the Bureau of Chemistry in 1907, show that the amounts of sugar which normally are present are usually increased materially by plucking the ears as they are formed.

Fermentable material in cornstalks.

Date.	Ears unplucked.			Date.	Ears plucked.		
	Sucrose.	Invert sugar.	Total sugars as invert sugar.		Sucrose.	Invert sugar.	Total sugars as invert sugar.
1907.	*Per cent.*	*Per cent.*	*Per cent.*	1907.	*Per cent.*	*Per cent.*	*Per cent.*
August 28	3.31	3.79	7.26	August 28	5.21	3.41	8.88
September 12	5.70	2.54	8.52	September 12	4.05	2.21	6.46
September 18	5.70	5.05	11.03	September 18	8.70	5.80	14.93
September 24	6.63	2.40	9.36	September 24	9.47	2.33	12.27
October 3	8.81	3.09	12.35	October 3	9.82	3.03	13.34

In 1906 Mr. Given was sent from the Bureau of Chemistry to cooperate with certain packers of corn in Hoopeston, Ill., in determining the value of corn waste as produced at a cannery. It was found that, roughly speaking, the wastes amounted to 40 per cent of the total weight of corn brought to the factory, not counting the husks as available, though these amount to about one-third of the total weight of the corn. As the result of a long series of tests on the various waste products, it was found that it was possible to produce from them from 6 to 10 per cent of alcohol, with a safe average of 8 per cent, or a yield of 3.2 per cent of alcohol calculated on the total weight of corn hauled to the factory.[a] On account of the expensive machinery and other apparatus required in the manufacture, it is hardly possible that a small factory could engage in the alcohol business, but a large factory could do so with apparent profit, or, where several factories were located within a short radius, by shipping their wastes to a central plant they might be utilized to advantage.

Sugar cane.—Under ordinary conditions sugar cane is too valuable a material to be used in the manufacture of industrial alcohol, but at times, as, for example, when canes have been heavily frosted, it may

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[a] This corresponds to 10.8 gallons of 180° alcohol per ton of corn hauled to the factory, or to 27.0 gallons per ton of waste at factory, excluding husks.

be as profitable to make them into alcohol as into sugar, providing that suitable facilities for distillation are available.

Sugar cane will yield anywhere from 20 to 40 tons per acre, the average crop in Louisiana being about 25 tons per acre. The percentage of sugar varies between 5 and 20 per cent.

When pressed between heavy rollers the cane yields from 60 to 65 per cent of its weight in juice on the first pressing, and from 7 to 12 per cent more on the second pressing. It may be presumed that 72 per cent of the total weight of the cane may be obtained in the form of juice by two pressings, leaving 28 per cent of the weight of the cane in the bagasse. The composition of this cane juice varies quite widely, according to the locality and the season. Spencer gives as the result of about 40 juice analyses made at Magnolia Plantation, Louisiana, the following average:

	Per cent.
Brix	16. 4
Sucrose	14. 1
Reducing sugars	5. 6
Total sugars calculated as dextrose	15. 4

On the assumption that 72 per cent of the cane can be obtained in the form of juice, that the juice contains 15.4 per cent of sugar calculated as dextrose, and that 13.3 pounds of dextrose will yield a gallon of alcohol at 180° proof, we should obtain from a ton of cane 16.7 gallons of alcohol. If cane is worth at the factory from $3 to $3.25 per ton, this would make the cost of the raw material for the alcohol about 19 cents per gallon.

Fruits.—Most fruit juices are rich in sugar, varying in content up to 30 per cent. They are much used at the present time in the manufacture of wines and brandies, but as the value of these liquors depends upon their flavors the fruits used in their preparation must be sound. In the preparation of alcohol which is to be denatured, the flavor is of no importance, and it has been presumed, therefore, that overripe or partly rotted fruit might be utilized as a raw material for industrial alcohol. This belief, however, is likely not to be confirmed by practical results, since satisfactory fermentation does not take place save in clean and reasonably sterile media. Furthermore, the supply of fruit is apt to fluctuate, so that a distillery depending upon waste fruit alone for its supply of raw material could not be sure from day to day, and still less from year to year, that a definite quantity would be provided.

The average amounts of sugar contained in different fruits and the quantity of alcohol that they might be expected to yield are given in the following table, the figures in which assume that a gallon of alcohol at 180° will be produced from 13.3 pounds of sugars, calculated as dextrose.

Theoretical yields of alcohol from various fruits.

Kind of fruit.	Average percentage of total sugars, calculated as dextrose.	Gallons of 180° alcohol yielded by total sugars contained in 1 ton of material.	Kind of fruit.	Average percentage of total sugars, calculated as dextrose.	Gallons of 180° alcohol yielded by total sugars contained in 1 ton of material.
Apple	12.2	18.3	Pear	10.0	15.0
Banana	13.8	20.7	Pineapple	11.7	17.6
Grape	15.0	22.6	Prickly pear	4.2	6.3
Orange	5.4	8.1	Tomato	2.0	3.0
Peach	7.6	11.4	Watermelon	2.5	3.7

It would be practically impossible to secure all of the sugar from any of these fruits in a condition suitable for fermentation, and therefore the actual yields of alcohol would always be lower than the theoretical yields given in the table. Thus, in the case of apples it would be impracticable to obtain more than 75 per cent of the total fermentable material, or about 9 per cent referred to the weight of apples as delivered. The actual yield of alcohol would be, therefore, about 13.7 gallons per ton, instead of 18.3. At $4 per ton for apples— a price which will fairly cover the expense of collecting culls and delivering them at the mill—the raw material will cost about 29 cents per gallon of alcohol. In the case of watermelons, it may be assumed that the expenses of gathering and delivery would not be less than $2 per ton. Assuming that as much as 4 gallons of alcohol could be obtained from a ton, the raw material would represent a cost of 50 cents per gallon. It is obvious that waste of this sort can not be converted into alcohol except at a loss.

Beet molasses.—This material, a refuse from the manufacture of beet sugar, is used in large quantities abroad, and to some extent in this country also, as a raw material for the production of alcohol. It usually contains very nearly 50 per cent of sucrose, with only traces of reducing sugars, and generally needs only to be diluted to prepare it for fermentation. One ton should yield between 75 and 80 gallons of alcohol at 180° proof. At $15 per ton this represents a cost of about 19 to 20 cents for raw material per gallon of alcohol.

Cane molasses.—This substance is very largely used the world over as a raw material for alcohol manufacture; and, so far as ease of manipulation is concerned, it unquestionably surpasses any other known material. Also, in the past it has been the cheapest material available. Ordinarily it contains both sucrose and reducing sugars in varying proportions. Such grades as have been used by distillers usually contain between 55 and 60 per cent of total sugars, calculated as glucose. A ton of such molasses, having a volume of approximately 170 gallons, should yield between 80 and 90 gallons of alcohol at 180° proof.

A few years ago such molasses was to be had at a price of about 2 or 3 cents per gallon, but at present—on account of its use as an ingredient in stock feeds—its value is from 9 to 12 cents per gallon. The amount required to produce a gallon of alcohol at 180° proof will cost on an average about 22 cents.

STARCHY MATERIALS.

This group includes those materials which contain an essential part of their fermentable matter in the form of starch, even though some of them, like artichokes and sweet potatoes, also contain material proportions of sugars. Its members differ from those of the preceding group in that they require a preliminary treatment, known as mashing, to convert their starch into a fermentable sugar. This process of starch conversion is chiefly effected by a ferment obtained from malt, and is fully described in the sections on ferments and mashing. (See pages 32 and 43.)

Jerusalem artichoke.—This root, though rarely used as a source of alcohol even in Europe, seems worthy of more attention than it has received as yet. It contains from 16.9 to 17.8 per cent of fermentable matter in the form of inulin and levulose, and as the inulin may be converted into levulose without the use of malt (by merely boiling under pressure) it should be worked very cheaply. With 17.5 per cent of fermentable substances it ought to yield about 28 gallons of strong alcohol per ton. It can be raised and delivered for about $5 per ton, so that the raw material for a gallon of alcohol should cost about 18 cents.

Cassava.—The root of the sweet cassava also is a promising raw material. The mean of a large number of analyses made in this Bureau shows it to contain about 65 per cent of moisture and 23 per cent of starch and sugars. A ton of root having this composition should yield about 38 gallons of alcohol at 180°. At $5 per ton, which would be a fair price with the modern methods of cultivation, the raw material would cost about 13 cents per gallon of alcohol.

Potatoes.—These vegetables offer, in regions where large quantities of culls are to be had, a very desirable material for alcohol production. With an average starch content of 18 per cent they should yield about 29 gallons of alcohol per ton. Assuming $4 to be a fair price for sorting and delivering a ton of culls, the cost of raw material for a gallon of alcohol will be about 14 cents.

Sweet potatoes.—These roots contain a considerably larger amount of fermentable matter than the common potato, much of it in the form of sugar; but, on account of their irregular form and the difficulty of removing the culls save by hand labor, they would probably be a more expensive material. With a content of 26.8 per cent of fermentable substances 1 ton should produce about 38 gallons of

alcohol, costing for raw material about 21 cents per gallon at a rate of $8 per ton.

Barley.—This grain, under present conditions, is too expensive to be used as a source of alcohol, save in the preparation of malt. It contains about 65 per cent of fermentable matter; and at a weight and cost per bushel of 48 pounds and 65 cents, 1 ton should produce about 98 gallons of alcohol, at a cost of 28 cents per gallon for the material.

Maize.—This material is, and for many years has been, the chief raw material for alcohol production in the United States. Dry corn of good quality should contain at least 65 per cent of sugars and starch, and should yield from 98 to 105 gallons of 180° alcohol per ton (shelled). At 56 pounds and 70 cents per bushel it represents a cost of about 25 cents per gallon of alcohol.

Oats.—This grain is not very well suited to distillery use, because of the glutinous nature of the mixture which is formed when it is treated with hot water. It contains about 50 per cent of fermentable substance, and might be made to yield about 70 gallons of alcohol per ton. At 32 pounds and 35 cents per bushel the raw material for a gallon of spirit would cost about 31 cents.

Rye.—This material is very largely used in distilleries which produce certain types of potable spirit, or compressed yeast; and it also is used in relatively small amounts in the yeast mashes of alcohol distilleries. It is not suited to use as the chief ingredient of the mash in an alcohol distillery, on account of the tenacious quality of the mash which it forms. Furthermore, it gives a low yield in proportion to the amount of starch which it contains. Though it usually contains over 60 per cent of fermentable matters, it rarely produces over 85 gallons of alcohol to the ton. At 56 pounds and 80 cents per bushel this represents a cost of about 34 cents per gallon for raw material.

Wheat.—What is said regarding the yield of alcohol to be obtained from rye probably applies equally well to wheat. The latter grain has not been much used in distilleries, even in the past when it was relatively cheap. At present prices it is hardly available as an alcohol material.

FERMENTATION PROCESSES.

CLASSIFICATION OF ENZYMS AND THEIR ACTION.

Since the success of nearly all operations directly connected with fermentation industries is dependent on enzymotic activity, a short discussion of the subject can not well be avoided in connection with a treatise on the manufacture of alcohol.

Enzymotic activity is of a katalytic character and for this reason a very small amount of enzym can produce a comparatively great proportion of chemical change. There are, however, enzyms whose activity is somewhat different, notably the oxydases; but in general it may be said that enzymotic action is hydrolytic; that is, it consists in splitting off simpler substances with a simultaneous absorption of water.

Enzyms differ greatly as regards the chemical reactions which they are capable of causing, and because of this fact they may be divided into several classes. From a practical point of view, the following subdivision of the enzyms is very satisfactory.

CARBOHYDRATE ENZYMS.

This group is characterized by its power to hydrolyze certain carbohydrates, and includes diastase, maltase, and invertase.

Diastase.—This enzym is distinguished by its ability to convert starch into maltose and dextrin, and this conversion has been practiced in the manufacture of beer and distilled spirits since very remote times. But it is less than a century since a definite substance found in the sprouting grain has been recognized as the actual converting agent. The change which takes place in soluble or gelatinized starch through the action of diastase is complex and proceeds by no means instantaneously. A considerable number of intermediate products are formed during the diastatic conversion of starch. The end product is maltose, but the conversion never proceeds quantitatively. For this reason the final product obtained in practice is a mixture of maltose and dextrin and the relative amounts of the two depend upon the conditions governing the conversion. In good practice, about 80 per cent of maltose is formed and about 20 per cent of dextrin, a

state of equilibrium being reached beyond which it is impossible to carry the original conversion. For this reason the diastase should not be injured in the mashing process, for it is due to its presence in the fermenting mash that as the sugar is consumed by the fermentation the dextrin is gradually converted into maltose, effecting practically a complete utilization of the starch in the original grain. Diastase, in converting starch into maltose and dextrin, exerts two distinct forms of activity—liquefaction and saccharification. It is known that the intensities of these activities depend largely upon the temperature at which the reaction takes place, and that the two types of transformation are differently affected by alterations in temperature. The liquefying power of diastase is exerted most strongly at 70° C., and is not wholly destroyed short of 93° C. The saccharifying power is strongest between 50° and 55° C., is weakened seriously at 63°, and is destroyed completely at 80°.

Maltase.—This enzym is rather widely distributed in the animal and vegetable kingdoms, being ordinarily classed with those enzyms known as glucases. The maltase of the yeast possesses the power of converting maltose into dextrose, and, since the presence of dextrose is only indicated in traces in fermenting maltose, it is presumable that the action of the maltase proceeds for the most part only within the yeast cell itself. It is very probable that the fermentation of maltose is due to its conversion into dextrose by this enzym. The optimum temperature for its action is 40° C., it is weakened very greatly at 50°, and is rendered inactive at 55°.

Invertase.—Invertase is capable of converting cane sugar or sucrose into invert sugar. This rather resistant enzym may be readily extracted from the yeast by various means. From yeast cells which have been killed with chloroform it may be extracted with water, and is precipitated from a water solution by the addition of alcohol. This white precipitate is readily dissolved in water and possesses the property of inverting cane sugar or sucrose quantitatively. Invertase is an important enzym in the fermentation of molasses or any other substances containing sucrose. Invertase acts only in a slightly acid solution. The optimum temperature for its action is about 55° C., it is slowly destroyed at about 65° C., and immediately at 75°.

Most yeasts secrete both invertase and maltase, and will, therefore, ferment either maltose or sucrose. Some races, however, such as *Sacch. octosporus*, ferment maltose but not sucrose, and are considered not to secrete invertase. Other races, like *Sacch. marxianus*, ferment sucrose but not maltose, and apparently, therefore, do not secrete maltase. Still others, such as *Sacch. apiculatus*, ferment neither of these sugars, and are presumed to secrete neither enzym.

PROTEOLYTIC ENZYMS.

These enzyms cause a splitting up of proteins into simpler substances and are designated according to the degree of disintegration which they produce. In the fermentation industries they play two important rôles, namely, in producing malts and yeast foods. The latter process takes place in the process of mashing. Since the proteins are nondiffusible, their value as nourishment for the yeast depends on their being broken down into simpler, readily diffusible substances, and it is by enzymotic action, governed by suitable conditions, that this breaking down process of the proteins is accomplished. Malt and yeast peptase and tryptase are included in this group.

Peptase.—Peptase produces reactions with proteins, with the formation of peptones as end products.

MALT PEPTASE was discovered somewhat over a quarter of a century ago and its ability to dissolve fibrin and to form peptones was reported. According to Windisch and Schellhorn, only minute traces of a proteolytic enzym occur in unsprouted barley, the amount increasing during the sprouting process. In the drying of the malt, the enzym is injured but is not destroyed. The changes produced in the albumin of the grain by this enzym depend on the acidity and the temperature of the solution. At low temperatures the reaction is slow, but proceeds much further than at higher temperatures; small amounts of organic acids also are favorable to the reaction.

YEAST PEPTASE or ENDOTRYPSIN occurs in the yeast cell and is an extremely energetic tryptic enzym. Yeast extract allowed to stand at ordinary temperature loses rapidly its content of coagulable albumin as well as its ability to produce fermentation in the sugar solution, and contains considerable quantities of crystallizable decomposition products of the albumins, notably lucin and tyrosin. These changes are due to the activity of this tryptic enzym. It is not alone in the yeast extract that the peptase accomplishes the destruction of zymase, but, as will be noted later, this destruction of the zymase may occur in the living yeast cell with a consequent loss of fermenting power.

Tryptase.—Tryptase produces amids and amido acids as end products from proteins. Malt tryptase and yeast tryptase are two important enzyms in this class.

ZYMASE.

This enzym in reality forms a class by itself, in that it possesses the property of converting monosaccharid sugars into alcohol and carbon dioxid. In 1896, Buchner prepared an extract by subjecting the yeast to a pressure of about 50 atmospheres to remove as much

water as possible and then grinding with sharp quartz sand to break the cell walls. This mixture of yeast and sand was then subjected to gradually increased pressure and the extract received in ice-cold vessels. Researches which have been made on this yeast extract have thrown light on a number of important points in connection with the fermentation industries. As would naturally be expected, a preparation of this kind contains a great number of different substances. The presence in this juice of the enzym which Buchner named zymase, and which is the cause of alcoholic fermentation, overthrows to a great extent the older theories which regarded the actual cause of the transformation of sugar into alcohol and carbon dioxid as a vital process dependent upon the actual life activities of the yeast cell itself. The presence of endotrypsin, invertase, and a number of diastatic enzyms as well as of zymase itself may readily be determined in the extract, and as is true of most other enzyms the zymase has never been isolated and prepared in pure form. The power of the yeast extract to produce alcoholic fermentation falls off very rapidly on standing, especially at ordinary or slightly increased temperatures. At 41° C. zymase is apparently destroyed. If the fresh extract is dried at a low temperature in vacuum an albumin-like mass results which for a long time retains its power, apparently unaltered, of causing fermentation. The extract may also be precipitated with a mixture of alcohol and ether. The precipitate after being washed with ether and dried is relatively stable as regards its fermentive power.

Of great importance from a practical standpoint is the relation in the yeast of the endotrypsin and the zymase to each other. Under normal conditions these enzyms act independently of each other upon the respective substances which it is their function to render of value for the life and development of the yeast. When the development of the yeast is stopped by lack of nourishment, whatever the cause, as is the case with compressed yeast, and is therefore in a state of so-called starvation, the activity of these enzyms does not stop and there ensues apparently a struggle for existence between them. Which will suppress the other is, according to Delbrück, directly dependent upon the temperature. The proteolytic enzyms destroy zymase at temperatures exceeding 2° or 3° C. The endotrypsin in the latter case breaks up and destroys not only the zymase, but also the protoplasm itself, and thus causes the death of the yeast cell which under normal conditions it is its function to help nourish.

OXYDASES.

Reactions produced by enzyms of this group are of an oxidizing nature and, though their value in distillery work may not be apparent, still they are important because of the rôle they undoubtedly

play in the vital processes of the organisms. They are always present in grain, but their presence in the yeast extract has been disputed, even though they have been shown to exist in the living yeast cell.

LIPASES.

These enzyms produce reactions with the fats, causing formation of fatty acids and glycerin. The presence of glycerin in distillery beers is probably due to these enzyms, which have been shown to exist in the yeast cell and in yeast extract. Their value to the success of distillery operations is not readily apparent.

MALTING.

By J. A. Le Clerc, *Bureau of Chemistry.*

As has been explained, the conversion of the starch of such raw materials as cereals and potatoes into a fermentable sugar is accomplished through the action of various enzyms, which almost invariably are obtained from the material called malt. This substance is a cereal product which may be obtained from any suitable grain by allowing the latter to sprout under carefully regulated conditions. The enzyms are not present in the grain originally, but are developed during the sprouting process; and, when once formed, they may be preserved for a considerable time, without any essential loss of strength, by carefully drying the malt. Any grain may be used for malting, but, though rye, oats, and corn are all treated thus at times for distillers' use, barley is almost universally employed, since it is more easily handled and gives a much larger yield of diastase.

TYPES OF MALTING BARLEYS AND THEIR COMPOSITION.

There are several types of barley, among which the following are important from the maltster's standpoint:

(1) a *Hordeum distichum,* a two-row type which includes the well-known varieties Chevalier, Hallett, Hanna.

  b *Hordeum zeocritum,* two-row fan-shaped barleys of which Goldthorpe is the leading variety.

(2) *Hordeum vulgare,* the ordinary six-row barley, such as Manchurian, Oderbrucker, Scotch, etc. Although these barleys have all three flowers on a spikelet fertile, and are really six-row, they are sometimes called four-row on account of their rectangular appearance when the heads are examined from the top, owing to the fact that the ears are narrow and that the grains overlap each other. In this respect they differ from the following variety:

(3) *Hordeum hexastichum,* which likewise have their flowers on a spikelet fertile, but on account of the fact that the ears are wide the appearance of the head is a hexagon when examined from the top. These are really six-row barleys. An example of these is the white barley of Utah and adjoining States.

The average composition of barleys of these three types,[a] together with data as to weight, are given in the following tabulation:

Average percentage composition of three types of malting barleys (Le Clerc).

Type of barley.	Moist-ure.	Water-free basis.						Weight per bushel.	Weight per 1,000 grains.
		Ash.	Pro-tein.	Fat.	Fiber.	Pento-sans.	Starch.		
								Pounds.	*Grams.*
Two-row.................	8.9	2.9	11.6	2.0	5.2	8.4	59.1	52	38
Ordinary six-row........	8.7	3.0	11.9	2.0	5.8	9.6	58.9	47	27
Hexastichum............	8.5	2.9	10.0	2.0	5.8	9.0	59.9	48	38

The two-row barleys are chiefly grown in Europe, although they are raised in this country in Montana, Idaho, New York, etc., to a limited extent. On account of their relatively high carbohydrate content and low protein, they are particularly adapted to use in the brewery.

In this country, the ordinary six-row barley is the kind most abundantly produced, being raised extensively in the States of the Mississippi Valley. Its relatively high protein content causes it to produce a malt of high diastasic power, and thus fits it especially to use in the distillery.

PHYSICAL CHARACTERISTICS.

A good distillers' barley should be free of dirt and have good odor and color, small grains of uniform size, a high percentage of nitrogen, and high germinating capacity. With these characteristics and with proper treatment in the malt house, it is bound to yield a malt of good character.

Cleanliness is necessary, not only because dirt is not a source of alcohol, but because it is sure to carry large numbers of bacteria and molds, which interfere with the production of a good malt. Good color and odor indicate immediately that the barley is free of mold and fungus growths. Uniformity in size indicates that the grains are of the same variety and age, and that they will germinate uniformly when subjected to the malting process. Richness in nitrogen, which usually is characteristic of a small-grained barley, promises an active malt, rich in diastase. Furthermore, the nitrogenous constituents of malt have great value as a yeast food.

Barley is composed of about 12 per cent husks, 10 per cent bran, 2.5 per cent embryo, and the rest endosperm or the stored food for the plantlet. The husks and bran are merely protective. The germ

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[a] U. S. Dept. Agr., Bureau of Chemistry, Bul. 124. Le Clerc and Wahl, Chemical studies of American barleys and malts.

is the seat of life; it consists of embryonic radicles or rootlets and the plumula or acrospire. The endosperm is composed mostly of starch and protein, but both of these substances are insoluble and non-diffusible and can not be used directly in supplying food for the young plant. The agency which renders these soluble and diffusible is an enzym or a series of enzyms secreted by the embryo during growth, one of which, the diastase, dissolves the starch and converts it into sugar and dextrin, while another, the peptase, acts on the protein. These enzyms are developed in the growing malt as the grain's need for food increases. The products of enzym action are soluble and diffusible, and can be directly used as food by the growing embryo.

Barley, to be considered good, should show a germination of at least 97 per cent. If below this limit of vitality, it should be reduced in price, or rejected. Grains which are incapable of germination are not only useless, but harmful, because they act as carriers of micro-organisms which may infect the rest of the grain.

MALTING OPERATIONS.

When a barley suitable for the purpose of malting has been obtained, it must be subjected to a number of operations before a malt can result. In the first place, it must be thoroughly cleansed by screening and washing, lime water being sometimes used. The screening removes the weeds, dirt, mold and fungus growths, and the adhering bacteria and spores, and also tends to separate grains of different sizes. The latter point is somewhat important, as in subsequent steeping the small grains would absorb water more readily than the larger ones and be ready for germination sooner, thus causing nonuniformity in growth. The barley is then treated with water, for a period varying from thirty to sixty hours, according to the temperature and the character of the water and of the barley.

The steeping tanks are usually made of iron, wooden vats being objectionable because they may absorb organic matter, susceptible of putrefaction, from the steep-water. The tanks are cylindrical and have conical bottoms which are provided at their points with outlet valves which permit the water to be drained off, or the barley to be discharged. They also are provided with overflow pipes and sometimes with devices for agitating and aerating the grain by the injection of air.

The water is changed rapidly at first, to complete the cleansing begun by the screening process, chaff and light grains being floated off together with microorganisms. If many of the latter are present, it may be necessary to add some mild antiseptic to the first wash waters.

When the washing is complete, the steeping proper may be said to begin. This process is necessary in order that the grain may absorb the water required for germination, and that certain extractive substances may be dissolved, which if not removed would injure the quality of the malt by giving it a bad taste and odor. The water is changed once a day, and the steeping may be considered properly done when the grain can be pressed lengthwise between the fingers without pricking the skin, the hull meantime becoming easily detached; or if when the grain is bitten the endosperm does not crack, or when upon cutting the grain the endosperm appears to have been wet throughout, with the exception of a minute speck in the center. Too long steeping may cause uneven germination, and sometimes the grain may not germinate at all, which is the case when it has been "dead steeped," or has become sodden. Too little steeping is better than too much, as water may easily be sprinkled on the floor, but it can not be so easily removed. The temperature of steeping should be below 60° F. The increase in weight of grain during the steeping process should be 45 per cent, and in the volume 25 per cent; four bushels of barley equaling five bushels when steeped.

After steeping the grain is germinated. The principal points to be observed in germination are to keep the grain moist, cool, and well aerated, to keep the malt floors scrupulously clean, and to avoid breaking the grains by rough handling. The grain is first spread out on a smooth floor, called the malting floor, in layers from 2 to 4 inches thick, for from ten to fourteen hours, for partial drying and aeration, and then heaped in the form of a "wet couch" from 6 to 20 inches thick, and allowed to germinate. The thickness of the heap depends on the temperature produced as the result of intracellular respiration, a high temperature necessitating a thin couch and vice versa. The couch is turned frequently so as to keep the grains uniformly moist, and to maintain an even temperature of approximately 60° F., a temperature best suited for the production of a high diastasic malt. After from twenty-five to thirty hours the grain begins to show visible signs of growth, as seen from the whitish appearance of the protruding radicle. The growing embryo having furnished the rootlets with food, they proceed to grow and appear as white spots. This process is called "chitting." The heap is then decreased to from 2 to 5 inches and is called a "floor" or "piece." The thickness here also depends on the temperature. The heat of the couch, floor, or piece, is caused by the respiration of the grain, an active chemical change taking place in consequence of the action of the enzyms on the starch and nitrogenous bodies, and carbon dioxid is given off as a product of starch decomposition. As the carbon dioxid produced is more or less injurious to the process, ventilation and aeration are necessary, and are pro-

duced by turning the piece occasionally. Frequent turning of the floor is also necessary to insure uniformity of temperature and of germination, care being taken, however, not to destroy the radicles during the process. Otherwise, grains with broken radicles may stop growing, or even die, and thus become a prey to bacteria, molds, etc. Sprinkling may be resorted to if the floor becomes too dry. The temperature should not exceed 68° F. at any time during germination, and this process should go on until the acrospire has attained the length of the grain, which requires from six to eight days, depending on the barley. During growth a fine cucumber-like odor should be noticeable, but if mold is produced this odor may be concealed. In properly germinated malt the color of the germ should not change, and the acrospire should develop uniformly in all kernels; the radicles should appear fresh and have grown together, forming a mat. The length of the acrospire in well-malted barley, made by this process, is from three-fourths to four-fourths of the length of the grain, the length of the radicles being about one and one-half times that of the grain.

It was thought at one time that the maximum diastasic power of malt was developed when germination had reached this state, but the contrary is now known to be true. When germination takes place at a low temperature, 54° to 57° F., there is a distinct increase in the amount of diastase up to the point when the acrospire is about twice as long as the grain. Between three and four weeks are required to reach this point, and a half more diastase is formed than in the short-grown malt. The product of this new method of manufacture is called long malt, and is far better for distillers' use than the other variety.

GREEN MALT.

"Green malt" contains about 42 per cent of water. It is so called to distinguish it from "dry" malt. This green malt may be used to advantage for the saccharification of starch, as it is very rich in diastase.

Although the use of green malt is more economical, because on drying more or less of the diastase is destroyed, and also because of the mechanical manipulation required, yet it is often of advantage and necessary to dry the malt as, for example, when it has to be transported and used subsequently. For that purpose malt is conveyed to the upper of the two floors of the kiln, where it is subjected at first to a temperature of about 95° F. for twenty-four hours, or until the bulk of the water has been driven off. The temperature is then gradually increased, and finally raised to from 122° to 131° F., until dried. This process is called kilning, the drying being necessary to remove all possibility of fungus and mold growth. The

final drying may be carried on even to from 145° to 176° F., at a loss, however, of more or less diastasic power.

Green malt has a higher diastasic power than kilned malt. The higher the temperature of kilning, the more diastase is destroyed; for example, green malt with a diastasic power equaling 100 will have, when dried at 122° F., a diastasic power of about 82; dried at 140° F., 78; and at 158° F. it is 53. These values in each case are referred to the dry substance of the malt.

COMPOSITION OF MALT AND THE ACCOMPANYING CHANGES IN THE BARLEY.

The composition of the malt and the various changes which have been produced in the barley by this process must be considered. Malt, though produced from barley, has an entirely different composition. From the moment when the barley was washed and steeped, preparatory to malting, to the end of the kilning process, the grain has gradually been changing in composition. In washing and steeping alone about 1.5 per cent of soluble substances have been removed; these consist of mineral substances, potassium, phosphorus, sodium, calcium, magnesium, and organic substances, such as sugar, soluble nitrogenous bodies, etc.

In the process of malting physiological changes have been produced. The cells first secrete cytase, which decomposes the cellulose of the cell wall, after which the action of diastase and peptase, respectively, renders the starch and protein soluble, the result being the growth of the embryo at the expense of the endosperm. This is shown by the appearance of the radicles and the acrospire. When fully malted, barley has undergone the following changes in composition, i. e., it has suffered a loss or a gain of most constituents, as follows:[a]

	Per cent.		Per cent.
Fat	− 7.7	Sulphur	+ 9.0
Fiber	− 8.4	Lecithins	+ 34.3
Pentosans	− 1.6	Hulls	− 8.5
Starch	− 28.0	Bran	− 37.0
Reducing sugars, as invert sugar	+400.0	Embryo	+ 78.7
Cane sugar	+ 71.0	Endosperm	− 10.2
Ash	− 20.7	Total protein	− 12.0
Potash	− 48.0	Soluble protein	+ 72.5
Lime	− 22.0	Soluble noncoagulable protein	+104.0
Magnesia	− 17.6	Soluble coagulable protein	+ 13.0
Phosphoric acid, total	− 12.7		

As will be seen, the barleys have lost on malting from 7 to 12 per cent of fat, fiber, nitrogen, and hulls, and from 20 to 37 per cent of starch, ash, and bran. On the other hand, the malting process has

[a] U. S. Dept. Agr., Bureau of Chemistry, Bul. 124, p. 74, Le Clerc and Wahl, average results for fourteen States.

increased some constituents as follows: Invert sugar, 400 per cent; cane sugar, 71 per cent; embryo, 70 per cent; and soluble nitrogen, 70 per cent. The increase in sulphur is due to the absorption of coal gas during the kilning.

O'Sullivan found that in the case of two samples of barley containing 0.9 and 1.39 per cent of cane sugar, respectively, the same when malted contained 4.5 per cent each, whereas maltose increased from 0 to 1.2 and 2 per cent, respectively, and glucose from 1 to 3 per cent. Most of the increase in cane sugar took place in the embryo, whereas the maltose is found chiefly in the endosperm. The increase in sugars came from the decrease in starch, which amounted to over 20 per cent.

No change in pentosans has been noted. The mineral constituents decrease as follows: Phosphorus, −13; potassium, −48; calcium, −22; magnesium, −17; the total ash, −21. Most of this loss was in the bran, which decreased 37 per cent. On the other hand, the embryo increased about 100 per cent, the gain being obtained at the expense of the other parts of the grain, namely, the hulls, bran, and endosperm. The total protein decreased 10 per cent, whereas the soluble nitrogen increased about 100 per cent, owing to the action of the peptase. During malting the carbonic acid (CO_2) produced by the respiration of the cells amounts to from 5 to 6 per cent, resulting mostly from the fat and sugar. The radicles amount to 4 per cent, and these are rich in mineral substances, particularly phosphorus and potassium, and in nitrogen. During storage, a slight loss due to the giving off of carbonic acid in the vital process is constantly noted. This, however, is smaller the less water the grain contains.

Barley is sold at 48 pounds per bushel and malt at 33 pounds per bushel. It happens, therefore, that, notwithstanding the loss in the process of malting, a maltster has more bushels of malt than the original number of bushels of barley. Besides this, malt on standing, or during transportation, almost invariably increases in weight owing to the absorption of water. One hundred parts of barley will yield the following proportions of products named: Steeped barley, 148 parts; green malt, 140 parts; kiln-dried malt, 76 parts; stored malt, 78 parts; sprouts, 4 parts; and loss from skimming, 1 part.

PREPARATION OF MATERIAL FOR FERMENTATION.

SACCHARINE MATERIALS.

As the saccharine raw materials contain their fermentable matter in the form of diffusible sugars which are already in solution, they require relatively little preliminary treatment to fit them for fermentation. Succulent materials, like sugar beets and fruits, are

best prepared by rasping or grinding and pressing. Fibrous materials, like sorghum and cane, may be pressed in roller mills or they may be sliced and extracted in diffusion batteries. Sugar beets also can be extracted by diffusion. Care should be taken not to cook any material which may tend to form a jelly, such as apples or beets.

A certain degree of acidity is desirable in any saccharine solution which is to be fermented, in order to inhibit the growth of bacteria which might interfere with proper development and activity of the yeast. Fruit juices in general are already sufficiently acid, but beet juice requires the addition of as much sulphuric acid as will produce an acidity of 0.16° to 0.18°. (See page 71.)

Beet and cane molasses need only to be dissolved in water, with the addition of enough acid to overcome the alkalinity of the raw material. Beet molasses, which contains about 50 per cent of sugar, should be diluted with three or four times its own volume of water, and the resulting solution should be brought to an acidity of 0.3° to 0.4° by the addition of sulphuric acid. Cane molasses, which contains about 60 per cent of sugars, may be diluted with four or five times its volume of water or with a mixture of water and sour spent beer reserved from a preceding distillation.[a] This liquid contains certain nitrogenous materials which stimulate the fermentative activity of the yeast; and the lactic acid which always is present in it checks the development of bacteria in the molasses solution. If spent beer is not to be had, as at the beginning of a season, sulphuric acid may be used for acidification, and ammonium sulphate may be added to stimulate the yeast in the proportion of 1 pound for every 50 gallons of molasses.

MASHING OF STARCHY MATERIALS.

The term "mashing" is used to designate the series of processes by which starchy raw materials are prepared for yeast production or fermentation through the application of heat and of enzymotic action. These processes include scalding or cooking and saccharification and result primarily in the conversion of the insoluble and unfermentable starch into a soluble and fermentable sugar and incidentally in the development of certain nitrogenous substances which are assimilable by the yeast and necessary to its activity.

The enzym or ferment which converts the starch into sugar is known as "diastase" and is obtained from malt, which therefore must be used in conjunction with such other starchy materials as are to be mashed. As is mentioned in the section on enzyms, diastase manifests two forms of activity—namely, liquefying and

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[a] In molasses distilleries this material is called "still-returns." In grain distilleries it goes by the name of "slop."

saccharifying, which are differently influenced by changes of temperature. The latter and more important form is at its maximum in the neighborhood of 50° to 55° C., is diminished very seriously at 62° C., and is destroyed entirely at 80° C. The somewhat less important liquefying power of diastase is, on the other hand, strongest at 70° C., and is not entirely destroyed below 93° C. . These facts determine the temperature limit below which the saccharifying process must be carried on.

THE OPEN MASH TUB.

The simplest mashing process is that which is employed in distilleries which work no other materials than rye and malt, and is
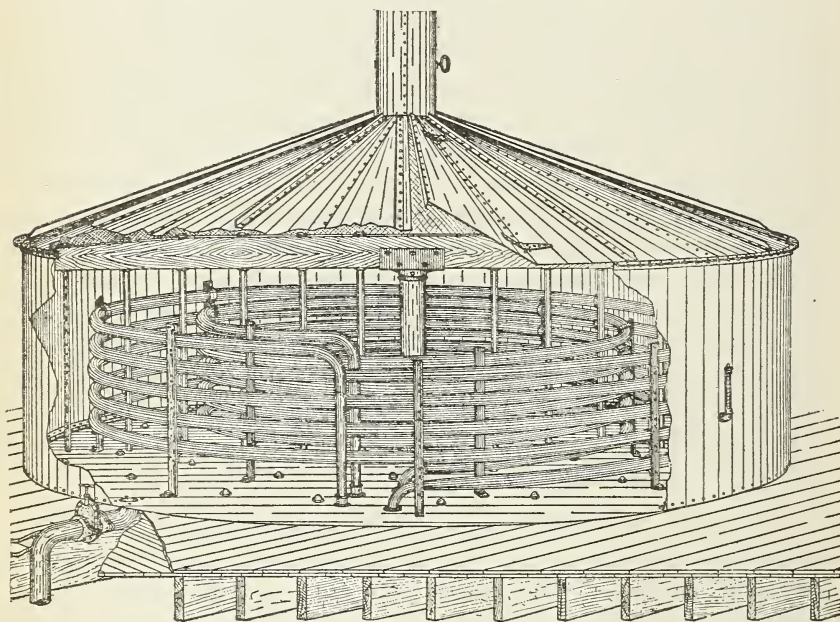
FIG. 1.—Open mash tub.

carried in an open mash tub (fig. 1), which usually is provided with a stirring rake, thermometers, inlet valves for steam, and coils for heating and cooling. The procedure of mashing is as follows:

Water at a temperature of 80° to 120° F. is run into the tub in the proportion of about 20 gallons for every bushel of grain to be mashed. The mixture of rye and malt, finely ground and containing from 10 to 20 per cent of the latter material, according to its diastasic capacity (pp. 118 and 119), is run slowly into the water, while the rakes are revolved fast enough to prevent the formation of clumps. As soon as the meal is all in, steam is admitted to the coils and inlets, and the temperature of the mash is raised (during the course of thirty to forty minutes) to 150° to 152° F., at which point it is held for an equal length of time, during which the starch conversion takes place. The mash is then cooled to about 70° F. and is pumped into the fermenting tubs, where it is mixed with yeast and more water.

When corn is to be mashed, in addition to or instead of rye, the process just described is not efficient and demands some modification. This is because the starch granules of maize, which are very largely embedded in a horny cellular tissue, demand a temperature considerably above 150° F. for their gelatinization. Accordingly the corn meal, mixed with about one-tenth of the total amount of malt,[a] is dropped into water at 120° F., and the temperature is raised as rapidly as possible to 212° F., where it is held for about fifteen minutes to allow time for gelatinization to be completed. Thereupon the mixture is cooled rapidly, and if rye is to be included in the mash it is added when 160° F. is reached. This temperature is maintained for about thirty minutes, after which it is reduced to 145° F., at which point the balance of the malt is added. If rye is not to be mashed, the temperature is reduced at once from 212° to 145°. In any case, the mash will be left at the latter temperature between thirty and forty minutes after the addition of malt for saccharification to take place.

VACUUM COOKER.

A small proportion of the starch of maize is inclosed so fast in the horny tissue that it resists saccharification even when the modified mashing process is employed. If it is planned to saccharify these last traces of starch, the open mash tub must be replaced by the so-called "vacuum cooker," in which the preliminary cooking of the meal is carried on under pressure. Such a cooker is represented in fig. 2. It is a horizontally placed cylindrical vessel made of steel plates and having a capacity of about 40 gallons for every bushel of grain to be mashed in one charge. Just below the center of each end it is provided with stuffing boxes through which passes a shaft that carries a pulley outside the shell and stirring arms, known technically as rakes, inside. In addition, thermometers and try cocks are mounted in each end. At the middle of the top of the cooker is a dome fitted with a pipe connection leading to a "cross." From one side of the cross connection is made with the steam supply, which must be carried at least at 50 pounds pressure; from the other side connection is made with the vacuum pump; and from the top a pipe is led to a blow-off valve. At one end of the cooker, and on top, there is a manhole through which water and meal are introduced. From the bottom of the shell, at one end, a discharge pipe provided with a valve leads to the drop tub. The operation of this cooker is as follows:

Water in the proportion of 20 to 25 gallons to a bushel of grain is first run in and warmed up to about 120° F. The rakes are then started, and the grain, which should be coarsely ground, is added at a rate slow enough to prevent lumping. When the grain is all in the cooker is closed, and the steam turned in. The temperature should be brought to the boiling point with the blow-off valve open. When this temperature is reached the blow-off valve is closed, and the pressure allowed to rise within the cooker at such a rate that the thermometers on the sides and the pressure

[a] Addition of the small proportion of malt at this point aids very materially in the gelatinization and liquefaction of the maize starch. Even though the diastase of this malt is destroyed, its use in this manner is distinctly worth while.

gauge in the top of the cooker indicate approximately a constant equalization of temperature. In corn distilleries the pressure is allowed to rise to from 50 to 55 pounds.[a] When the desired pressure is reached it is maintained for a very few moments only. The steam is then turned off, the blow-off valve carefully opened, and the pressure within the cooker completely released. The blow-off valve is again closed, and the valve connecting the cooker with a powerful vacuum pump is opened. By means of this vacuum the contents of the cooker are rapidly cooled to such a point that the addition of the necessary malt will further reduce the temperature to between 140° and 144° F.

The malt used represents ordinarily from 8 to 15 per cent of the grain forming the charge. It is ground relatively fine and is prepared for addition to the cooker charge
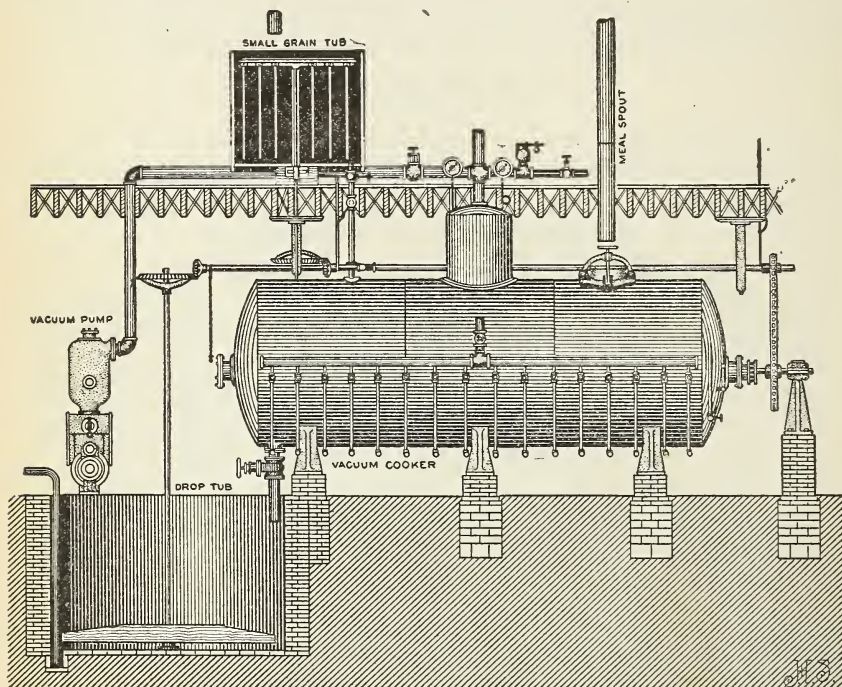
FIG. 2.—American vacuum cooker.

by thorough stirring with water, usually cold, in the small grain tub provided for this purpose. Sufficient water is used to allow the malt to flow easily through the piping from the small grain tub to the cooker. The length of time allowed for conversion in the cooker varies from fifteen minutes to one hour, but it is advisable not to hasten this part of the operation unduly, and the completeness of the conversion should always be checked by testing before pumping the mash to the fermenters.

HENZE COOKER AND MASH TUB FOR POTATOES.

It has been found in Germany, where potatoes rather than grain are the chief source of alcohol, that the mashing apparatus used in this country and described in the preceding pages is unsuited to

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[a] Corresponds to a temperature of about 300° F.

handle any material but grain. Accordingly, radically different mashing machinery has been devised abroad for the treatment of potatoes. This type of cooker may either be conical or a combination of cylinder and cone (see fig. 3). It is made of heavy steel plate and is provided with a manhole in the top for filling and steam connections at top and bottom, each being fitted with a globe valve and a check valve. There is also a drainage cock for condensed water and an outlet valve (h) of peculiar form, which usually is a heavy steel casting. The way through this valve is constricted and
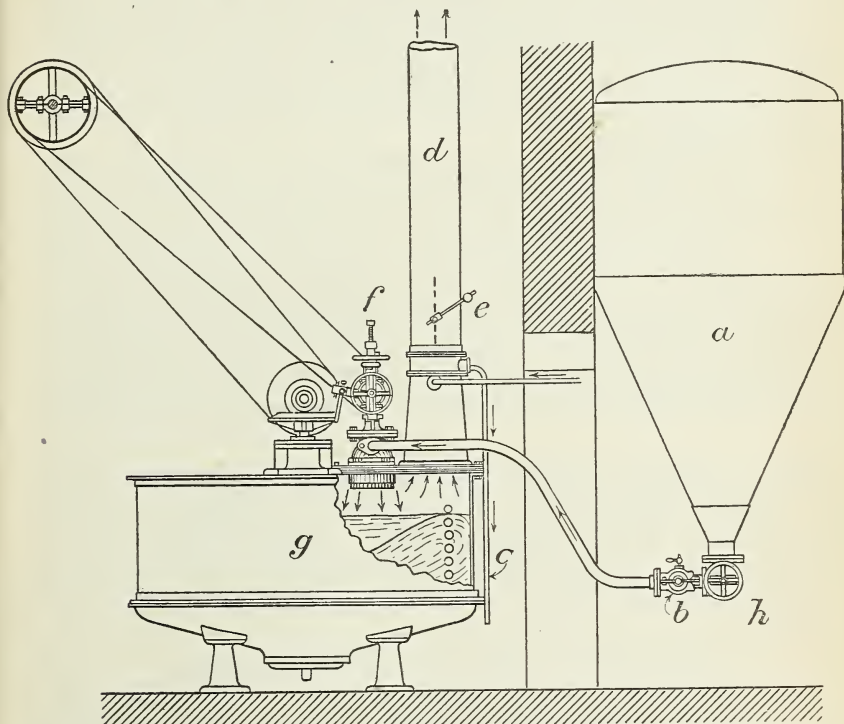
FIG. 3.—Henze cooker and German mash tub.

a, Cooker; b, stone catcher and discharge pipe; c, outlet for water condensed in ventilator; d, ventilator; e, damper; f, mash mill; g, mash tub; h, outlet valve and drainage cock.

lined with sharp ridges or teeth designed to shred the material passing through it. The apparatus should be tested to a pressure of about nine atmospheres.

The mash tub, like those used in the United States, is provided with a stirring rake and with coils through which cold water is made to circulate, but the speed of the stirrers and the ratio of cooling surface to mashing capacity are much greater than in apparatus of the American type. Sometimes a grating which serves as a stone catcher and a mash mill which completes the comminution of the

mash, are inserted in the pipe leading from the cooker to the mash tub. The details of operation are as follows:

Potatoes which are to be mashed in this apparatus must first be freed of dirt in a mechanical washer, after which they are weighed and placed, whole, in the cooker through the manhole. All the openings of the cooker are then closed, with the exception of the drain cock, and steam is allowed to enter slowly through the top connection. The condensed water which is formed while the charge is heating up is allowed to escape freely until the potatoes are thoroughly heated and steam escapes from the drain. This preliminary heating should take from fifteen to twenty minutes. The drainage cock is now closed and the admittance of steam at the upper connection is continued until the pressure gauge registers from 30 to 35 pounds. Thereupon, the lower connection is opened and the upper one is shut, and the pressure is allowed to rise to 45 or 50 pounds, at which point it is held for about fifteen minutes. The upper steam inlet is now opened again, the lower one being closed at the same time, and the outlet valve is opened and the whole charge is blown into the mash tub under full steam pressure.

While the preceding operations are going on, malt meal in the proportion of about 3 per cent of the weight of the potatoes, is mixed in the mash tub with a suitable amount of luke-warm water. When the softened potatoes are about to be blown into the tub the rake is set in rapid motion and a strong stream of cold water is turned into the cooling coils. The temperature of the mixture in the mash tub will usually be about 122° F. at the start, and should be allowed to rise gradually during the entrance of the cooker charge until at the end the temperature of 140° F. is reached. The amount of time required for emptying the cooker will depend somewhat on its capacity, about forty-five to fifty minutes being allowed for 6,000 pounds of potatoes. The maximum mashing temperature should be maintained for an equal length of time.

Grain, as well as potatoes, can be mashed in apparatus of this type without being ground. Water should be added in the same proportions as in the horizontal cooker. Some distillers consider it advantageous to add the malt in three portions—at the beginning, in the middle, and at the end of the mashing process.

THE YEAST AND ITS PREPARATION.

The yeast is a single-celled vegetable organism which, under the conditions normally prevailing in distillery practice, reproduces itself by budding or sprouting. It is possible for the yeast, under conditions more or less unfavorable to its development, to reproduce itself by the formation of spores, which, when the conditions become more favorable, develop in much the same manner as a seed develops in some of the higher forms of plant life and reproduces the original plant. As is the case with higher organisms, there are a number of organic and inorganic substances necessary for the growth and development of the yeast. Oxygen, nitrogenous and nonnitrogenous organic substances, and inorganic mineral substances are absolutely essential in yeast culture. These various yeast foods are present in abundance in all properly prepared yeast mashes.

KIND OF YEAST.

The various methods for the preparation and maintenance of distillery yeast vary much more in detail than in principle. The first requisite is a culture of yeast suitable for this special purpose.[a] This culture may be either biologically pure or it may be one obtained by spontaneous fermentation, in which the yeast that it is desired to propagate predominates. From every point of view the biologically pure culture has the advantage and is always to be preferred. It must be remembered that there are a great many varieties or types of yeast which are capable of producing alcoholic fermentation to a greater or less degree, and that in a mixture of perhaps a dozen different yeasts there will be but one race present which possesses the ability to produce the results desired. If now this race is isolated and propagated biologically pure, it is very evident that the results obtained will be much more uniform than those given by the original mixture, in which the yeast of desired character was only one of a large number, all of which were fighting for supremacy.

The scope of the present work will not permit of a discussion of the methods of preparation of pure cultures, and indeed it is seldom if ever that the small distiller would have the facilities or possess the technique necessary for their preparation. Assuming that a suitable culture is available, only its propagation and application in the distillery will be considered.

RAW MATERIAL FOR THE YEAST MASH AND ITS SACCHARIFICATION.

The raw material used in yeast making in grain distilleries consists of equal parts of rye meal and malt. In preparing the mash in which the yeast is to be grown, the requisite amount of water is run into the yeast tub and warmed to a temperature of from 60° to 70° C. The rye meal, which should not be too finely ground, is then slowly added with constant stirring, which is continued while the malt, previously ground, is run in. The temperature is then taken and should be in the neighborhood of 50° C. The stirring is continued and the temperature gradually raised during the course of an hour to 62° or 63° C. The mash is now allowed to stand at this temperature for at least two hours in order that the saccharification may be as complete as possible. The concentration after saccharification should not be less than 24° Balling, and 26° is preferable.

The next step in the preparation of the mash is the souring. This requires from eighteen to thirty-six hours and is effected by the activity of a suitable type of lactic acid bacterium which is allowed to grow in the mash until the desired degree of acidity is obtained. A mash left to itself at the proper temperature and for a sufficient

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[a] See pages 113 and 122.

length of time will sour spontaneously. But it is advisable, as in the case of pure yeast cultures, to start the souring with a pure culture of lactic acid bacilli whose ability to produce a clean lactic acid souring is known. Such cultures may be obtained from any fermentation laboratory.

SOURING OF THE YEAST MASH.

After the saccharification of the mash is complete the temperature is reduced to 55° C., and the culture of lactic acid bacteria is added and thoroughly stirred in. It is then allowed to stand, care being taken that the temperature does not fall below 50° C. At the end of a period of from eighteen to twenty-four hours from 1.8° to 2.1° of acidity[a] will have been produced, and this is ordinarily sufficient. A small amount of this soured mash is now removed, to be added to the next mash to start the souring.

As soon as the mash has reached the proper degree of acidity and the soured material has been removed to start the next souring, its temperature is raised to from 70° to 75° C. and held for from twenty to thirty minutes. This heating of the soured mash not only serves to kill the lactic acid producing organisms, whose further development is undesirable, but also serves to destroy or at least to render harmless a great variety of organisms whose activity might otherwise cause much trouble either in the yeast itself or later in the fermentation of the main mash. It is the purpose of the souring process to produce a medium that will be more favorable for the development of the yeast than for the development of bacteria or other undesirable organisms. Bacteria as a class are much more susceptible to the influence of small amounts of acids than are the yeasts, and it is this fact which is taken advantage of in the souring or acidifying of the mash in which it is desired to grow a vigorous yeast.

In the preparation of yeast mashes the bacteria which are the ordinary trouble makers are those producing acetic and butyric acid. The hay bacillus (*Bacillus subtilis*) which often develops in spots as a white skin-like growth on the surface of a mash seldom if ever causes trouble. Putrefactive organisms do not have an opportunity for development during the preparation of the mash, and indeed it may well be remarked that the extensive development of any organism, save the lactic acid bacteria and the yeast itself, is always due either to ignorance or to gross carelessness on the part of the operator. A mash which has been correctly soured possesses a very agreeable odor and a decidedly sharp acid taste.

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[a] See page 71.

FERMENTING THE YEAST.

After the mash has properly soured and heated, as described above, it is cooled as rapidly as possible by stirring and the circulation of water in the coils to 30° C., at which temperature the start yeast or the mother yeast, as the case may be, is added. The cooling is then continued down to from 15° to 18° C., depending upon conditions, such as size of mash, temperature of yeast room, ventilation, etc. The temperature rises very rapidly with the fermentation of the yeast, and unless properly controlled is apt to rise to such an extent as to injure the yeast. When the temperature has risen to 25° C., the mother yeast, with which it is intended to start the next batch, is removed and, if possible, placed immediately in the oncoming soured mash. If this is impossible, it is cooled to 15° C. and kept at this temperature until used. The temperature of the fermenting yeast should not be allowed to rise above 30° C., and the mash should be allowed to ferment while the saccharimeter reading decreases to about 5° Balling.

DEVELOPMENT OF THE START YEAST.

In the preparation of start yeast from biologically pure cultures, the latter are usually maintained in tubes or small bottles on wort-agar jelly. If kept cool and transplanted from time to time, such cultures may be kept in perfect condition indefinitely. The yeast produces a heavy creamy white growth upon the surface of or within the jelly, as the case may be, and will frequently crack the jelly completely into two or more sections.

The development of the yeast from the tube culture up to the point where it is used as start for the mash is accomplished in sterilized wort—that is, a sterilized filtrate from a sweet mash. For the preparation of a wort, a mash is prepared from malt and rye meal (3:1), as previously directed. This mash is not soured, however, but as soon as saccharification is complete it is thoroughly boiled and then strained, and the resulting wort filled into the necessary containers and sterilized. The containers vary in size from 10 cc. to 100 liters or more. Glass tubes and flasks are ordinarily used for the smaller containers, and suitable copper vessels for the larger. The vessels, especially the larger ones, are frequently provided with a tube extending to the bottom, by means of which filtered air may be blown through the wort during its fermentation to stimulate the growth and development of the yeast. The wort used should have a concentration of about 10° Balling. A small quantity of yeast is removed from the wort-agar culture by a looped platinum wire, using the ordinary bacteriological precautions to guard against infection, and placed in a tube containing about 10 cc. of sterile wort.

The tubes, flasks, etc., containing the wort are, of course, closed with cotton plugs before sterilizing. The tube is then allowed to stand at ordinary room temperature until in a state of vigorous fermentation. This will require usually from eighteen to thirty-six hours, depending largely upon the condition of the original agar culture. As soon as a vigorous state of fermentation is reached, the contents of the tube are transferred by pouring to a small flask containing about 80 cc. of wort. Fermentation is allowed to proceed in this flask until the yeast produced has settled largely to the bottom. The greater part of the supernatant liquid is then carefully poured off, and the remainder, containing the yeast, added to about 500 cc. of wort in a flask of 1 liter capacity. The 500 cc., when in a state of vigorous fermentation, may be added directly to about 5 liters of wort in a large flask or copper container. From 5 liters one may go to 50 liters, and from 50 to 500, and so on until sufficient has been obtained in sterile wort to be used as start in the first yeast mash proper.

In this line of work good results are obtained by using one volume of inoculating material to ten volumes of sterile nutrient material. After a start has been made it is not necessary to go back every time to the original tube culture in order to obtain fresh yeast. It is simpler to retain a sufficient quantity in sterile wort in a closed copper jug. Such cultures in suitable jugs or cans may be kept in good condition for a long time if stored in an ice chamber or in very cold running water.

POTATO YEAST.

In the case of potato distilleries, it is advisable, from the point of economy as well as for the sake of convenience, to use the potato mash directly from the mash tub, together with either dry or green malt, in the preparation of the yeast. The potato contains a great deal of material suitable as food for the yeast, and in this respect has the advantage over corn as used in grain distilleries, which contains very little material capable of assimilation by the yeast. The conditions governing the preparation of a potato yeast are similar to those already described. The mashing, souring, and fermenting of yeast mashes—in other words, the successful handling of distillery yeast—depend upon absolute cleanliness, attention to detail, and unabating watchfulness combined with good judgment.

DISTILLATION.

OPERATION OF A SMALL LABORATORY STILL.

Alcohol is separated from solutions and obtained in concentrated form by the process of distillation, which is carried on in an apparatus called a still. In actual construction stills are very complicated; but in their more elementary forms they are extremely simple both in principle and in structure.[a] The illustration (fig. 4) represents a small laboratory still and shows the essential parts found in the larger stills which will be discussed later. Such an apparatus as is shown in fig. 4 is operated as follows:

The liquid which is to be distilled is placed in A, and heat is applied beneath, the opening at B being closed either with a plain stopper or
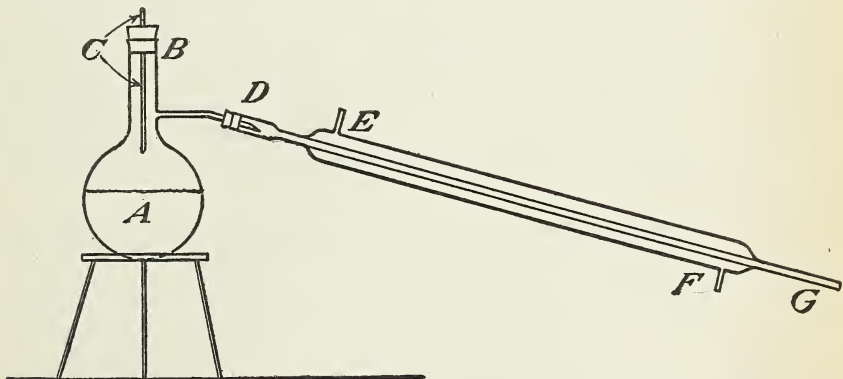
Fig. 4.—Small laboratory still.

A, boiler or kettle of still; B, opening for filling or charging; C, thermometer, sometimes omitted; D, connection to condenser; DG, inner condenser tube, passing through EF, water jacket, with water inlet at F, and outlet at E; G, outflow for distillate.

with a cork carrying a thermometer. The side neck at D is connected tightly with the end of a condenser, consisting of a long tube surrounded by a water jacket. Some time before the liquid in A begins to boil, a stream of cold water is introduced in the condenser jacket through the inlet F; and when boiling begins the vapors pass through the outlet D into the inner tube of the condenser, and on coming in contact with its chilled walls in the region EF are condensed—or reduced again to the liquid form—and flow out at G. These condensed vapors are called a *distillate*.

[a] See lecture on the history of distillation (p. 90) and early forms of stills shown in figs. 12 and 13.

THE THERMOLOGY OF DISTILLATION.

During such an operation the heat applied at A is first used up in raising the temperature of the liquid to its boiling point. When that point is once reached, continued application of heat will not raise the temperature further, but will convert the liquid into a vapor at the same temperature. The heat which is consumed in the first process and is accumulated in the hot liquid is called its "sensible heat." That which is consumed in the second process, seeming to disappear during the vaporization, is called "latent heat of evaporation." The amount of heat required for either process can be measured and stated in terms of any convenient unit. The unit employed in technical work in Great Britain and the United States is called the British thermal unit—abbreviated B. t. u.—and represents the quantity of heat which is required to raise the temperature of 1 pound of pure water 1° F.

It has been found that equal weights of different liquids require different amounts of heat to raise them through equal intervals of temperature, or to volatilize them from their respective boiling points. Thus, the heat consumed in raising the temperature of 1 pound of pure alcohol 1° F. is only 0.65 B. t. u. (approximate), or 65 per cent of that required to raise the temperature of an equal weight of water through the same interval. The ratio 0.65 is said to be the "specific heat" of alcohol.

In volatilizing a pound of water from and at its boiling point, 212° F., about 965 B. t. u. are consumed. In evaporating the same weight of alcohol from and at its boiling point, 173°, only 365 B. t. u. are consumed. The values 965 and 365 B. t. u. are said to be the respective "latent heats of evaporation" of water and alcohol.

When a pound of any liquid is distilled its vapors carry into the condenser a quantity of heat equal to its latent heat of evaporation plus the number of B. t. u. which were needed to raise the material from its initial temperature to the boiling point. This amount is called the "total heat of evaporation." With water, at an initial temperature of 60°, it is 1,117 B. t. u. With alcohol, at the same initial temperature, it is approximately 500 B. t. u.

In order for any part of the vapors to be condensed, a portion of this heat must escape into their surroundings. If the condensation is to be complete, a quantity equivalent to the latent heat of evaporation must escape; and if in addition the distillate is to be cooled to the initial temperature of the liquid which was distilled, the total heat of evaporation must be given off. Some condensation and cooling may be effected by loss of heat from the vapors to the apparatus and the surrounding atmosphere, but this action usually is very slight, so that the condenser is necessary as the instrument of heat exchange.

Heat is transmitted through the walls of the inner condenser tube to the water in the jacket, the rate of transmission depending chiefly on the difference between the temperatures at the inner and outer surfaces of the tube. The size of the condenser depends upon this rate, and on the volume of heat which must be exchanged in a given time. In order to secure the most effective cooling with a minimum amount of water, the "counter-current" principle is always employed, the water being circulated in the direction opposed to that of the hot vapors and distillate.

The weight of water used in cooling, multiplied by the rise in its temperature, measures in B. t. u. the amount of heat withdrawn from the vapors and distillate. Thus, if 1 pound of pure water is distilled, its initial temperature and that of the distillate being 60°, 1,117 B. t. u. must be removed by the cooling water. If 10 pounds of the latter are used, at an initial temperature of 55°, each pound must absorb 111.7 B. t. u., and the final temperature will be about 167°. If 1 pound of alcohol is distilled, instead of water, only 500 B. t. u. have to be disposed of, and less cooling water will be needed ($\frac{500}{112} = 4.5$ pounds, approximately). A recognition of the preceding facts is necessary in determining the efficiency of apparatus and in ascertaining the cost of manufacture.

When a mixture of alcohol and water is boiled in such a still as has been described, the following observations may be made:

(1) The mixture begins to boil at a temperature intermediate between the boiling points of pure alcohol and pure water; and there is a definite relation between this boiling point and the percentage composition of the mixture, liquids rich in alcohol boiling at lower temperatures than those that are not so rich.

(2) The vapors rising from a boiling alcoholic liquid are at any moment richer in alcohol than the liquid itself—save in the neighborhood of 95 to 100 per cent. There is a definite relation between the composition of any mixture and of its vapors.

(3) Since the vapors given off by a boiling alcohol solution are richer than the solution itself, it follows that the liquid grows continually weaker as distillation proceeds, and that the first portions of the distillate are stronger than those which come over subsequently. It follows also, that the boiling point rises as distillation proceeds.

(4) If a sufficient proportion of water is present in the original mixture, a time will come when the distillate will cease to contain alcohol and when the boiling point will reach 212° F. At this point nothing but water remains in the still. The distillate which has been collected, up to this point, contains all of the alcohol which was present in the original mixture, in the form of a solution stronger than the original one.

(5) In its early stages, when the distillate is richest in alcohol, such a distillation proceeds more rapidly than when the alcohol has all gone over—it being presumed that the supply of heat is uniform. In other words, a given amount of alcohol may be distilled more readily and economically from a strong solution than from a weak one.

On the foregoing facts are based our methods for obtaining strong alcohol by the process of distillation. The relations between the boiling points and the percentage compositions of alcohol solutions of different strengths, and the percentage compositions of the vapors rising from these solutions at their respective boiling points, are given in the following tabulation, which is based largely on the investigations of Sorel:

Table showing the relation between the strength of an alcoholic liquid, its boiling point at normal atmospheric pressure, and the strength of its vapors.

Alcoholic strength of liquor in still.		Boiling point (°F.).	Alcoholic strength of vapors (weight per cent).	Alcoholic strength of distillate (volume per cent at 60° F.).	Alcoholic strength of liquor in still.		Boiling point (°F.).	Alcoholic strength of vapors (weight per cent).	Alcoholic strength of distillate (volume per cent at 60° F.).
Volume per cent at 60° F.	Weight per cent.				Volume per cent at 60° F.	Weight per cent.			
0	0.00	212.0	0.00	0.00	51	43.42	181.3	68.17	75.27
1	.80	210.6	7.97	9.90	52	44.37	181.1	68.50	75.58
2	1.60	209.1	14.36	17.70	53	45.31	180.9	68.87	75.90
3	2.40	207.8	20.61	25.20	54	46.27	180.7	69.23	76.22
4	3.20	206.5	25.74	31.25	55	47.24	180.5	69.59	76.54
5	4.00	205.2	29.72	35.75	56	48.19	180.3	69.95	76.86
6	4.81	203.9	32.74	39.30	57	49.16	180.1	70.31	77.18
7	5.62	202.7	35.69	42.60	58	50.15	179.8	70.68	77.50
8	6.43	201.5	38.31	45.50	59	51.14	179.6	71.05	77.83
9	7.24	200.4	40.98	48.40	60	52.14	179.4	71.43	78.17
10	8.05	199.5	43.50	51.10	61	53.14	179.2	71.83	78.52
11	8.87	198.5	45.74	53.45	62	54.15	178.9	72.21	78.86
12	9.68	197.6	47.95	55.75	63	55.15	178.7	72.60	79.20
13	10.49	196.7	49.62	57.45	64	56.16	178.5	73.01	79.56
14	11.31	195.7	51.94	59.80	65	57.18	178.3	73.43	79.92
15	12.14	194.8	53.74	61.50	66	58.21	178.1	73.84	80.28
16	12.96	194.0	55.11	62.95	67	59.26	177.9	74.28	80.65
17	13.79	193.2	56.22	64.05	68	60.32	177.7	74.73	81.05
18	14.61	192.5	57.16	64.95	69	61.38	177.5	75.19	81.45
19	15.44	191.8	57.88	65.65	70	62.44	177.3	75.66	81.85
20	16.27	191.2	58.45	66.20	71	63.52	177.0	76.20	82.30
21	17.10	190.5	58.86	66.60	72	64.60	176.8	76.75	82.75
22	17.93	189.9	59.26	67.00	73	65.68	176.6	77.27	83.20
23	18.77	189.3	59.68	67.36	74	66.77	176.4	77.80	83.65
24	19.60	188.8	60.01	67.70	75	67.87	176.2	78.35	84.10
25	20.44	188.3	60.28	67.95	76	68.98	176.0	78.89	84.55
26	21.28	187.8	60.54	68.20	77	70.11	175.9	79.43	85.00
27	22.12	187.3	60.92	68.47	78	71.24	175.7	80.01	85.48
28	22.97	186.9	61.12	68.75	79	72.38	175.5	80.62	85.98
29	23.82	186.5	61.38	69.00	80	73.52	175.3	81.25	86.49
30	24.67	186.1	61.65	69.26	81	74.67	175.1	81.88	87.00
31	25.52	185.8	61.91	69.50	82	75.84	174.9	82.51	87.50
32	26.38	185.6	62.19	69.77	83	77.03	174.7	83.15	88.02
33	27.24	185.3	62.49	70.05	84	78.23	174.5	83.80	88.53
34	28.10	185.0	62.74	70.30	85	79.43	174.3	84.45	89.05
35	28.97	184.7	63.09	70.60	86	80.64	174.1	85.15	89.59
36	29.84	184.5	63.38	70.87	87	81.88	174.0	85.84	90.12
37	30.71	184.2	63.68	71.15	88	83.13	173.8	86.55	90.67
38	31.59	184.0	63.98	71.43	89	84.39	173.6	87.28	91.23
39	32.47	183.7	64.25	71.68	90	85.68	173.4	88.04	91.80
40	33.36	183.5	64.55	71.95	91	86.98	173.3	89.04	92.55
41	34.25	183.3	64.83	72.22	92	88.30	173.2	89.99	93.25
42	35.14	183.1	65.12	72.50	93	89.65	173.0	90.74	93.80
43	36.04	182.9	65.45	72.80	94	91.01	172.9	91.71	94.50
44	36.94	182.7	65.80	73.12	95	92.41	172.8	92.80	95.25
45	37.85	182.5	66.16	73.45	96	93.96	172.7	93.96	96.00
46	38.76	182.3	66.49	73.75	97	95.41	172.7	95.41	97.00
47	39.68	182.1	66.83	74.05	98	96.82	172.7	96.82	98.00
48	40.61	181.9	67.16	74.35	99	98.38	172.8	98.38	99.00
49	41.53	181.7	67.49	74.65	100	100.00	172.9	100.00	100.00
50	42.48	181.5	67.81	74.95					

The figures of the foregoing tabulation are based on the assumption that the alcoholic vapors are removed and condensed as soon as formed without any preliminary partial condensation inside the still itself, such as may be caused by radiation from its walls. In practice, however, radiation always occurs, resulting, as will be explained shortly, in a dephlegmation of the vapors which reach the condenser, and thereby in an increase in their alcoholic strength.

If an alcoholic liquid is redistilled, the concentration of which has previously been increased by distillation, its strength can be raised again, though within narrower limits than in the preceding case. Repeated distillations will continue to increase the concentration, but by increasingly smaller intervals, as will be seen by inspection of the foregoing table. This process of strengthening an aqueous solution of alcohol through the removal of part of its water by means of repeated distillation is known as rectification.

If the hot vapors which rise from a mixture of alcohol and water boiling under atmospheric pressure be partially cooled a part of the vapors will be condensed, the amount depending upon the extent of the cooling. The percentage of water in the condensed part, or *condensate*, will be greater than in the original vapor; and the percentage of alcohol in the vapors remaining uncondensed will also be greater than before, although the total amount of alcohol in vapor form may have been materially diminished. This process of increasing the concentration of alcohol vapors by means of partial condensation is called dephlegmation. It accomplishes the same result as rectification, though in an opposite manner. The two processes go on simultaneously in all alcohol stills, and the efficiency of the large modern stills which are employed in the production of high-proof alcohol is due to the perfection with which they maintain a balance between the two processes, the condensates being reboiled until by successive repetitions a mixture is separated into water and a nearly anhydrous alcohol.[a]

THE EARLIEST TYPE OF INDUSTRIAL STILL (FIRE POT STILL).

The fire-heated type of still first used in industrial distillation, and even now employed by distillers of French brandies, is identical in all essentials with the laboratory still just described. The construction of such an apparatus is shown in fig. 5, and it is operated as follows:

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[a] It is impossible, on account of the formation of an alcohol-water mixture of minimum boiling point, to produce an alcohol stronger than about 97 per cent by volume, even in the most perfect stills.

OPERATION.

The kettle is charged with about 264 gallons of wine (1,000 liters), containing approximately 10 per cent of alcohol, and a fire is kindled on the grate, wood or a long-flame coal having preference as fuel. About three hours are consumed in heating the charge to the boiling point—approximately 200° F.—as the transmission of heat in such an apparatus is very slow. The distillate, when it begins to come over, contains from 60 to 70 per cent of alcohol, and is collected in one portion so long as it contains any spirit. The entire operation lasts nine or ten hours, and the product is about 85 to 90 gallons of a liquor containing approximately 30 per cent of alcohol. The exhausted liquor remaining in the kettle is then run to waste, and the apparatus is recharged for a second operation.
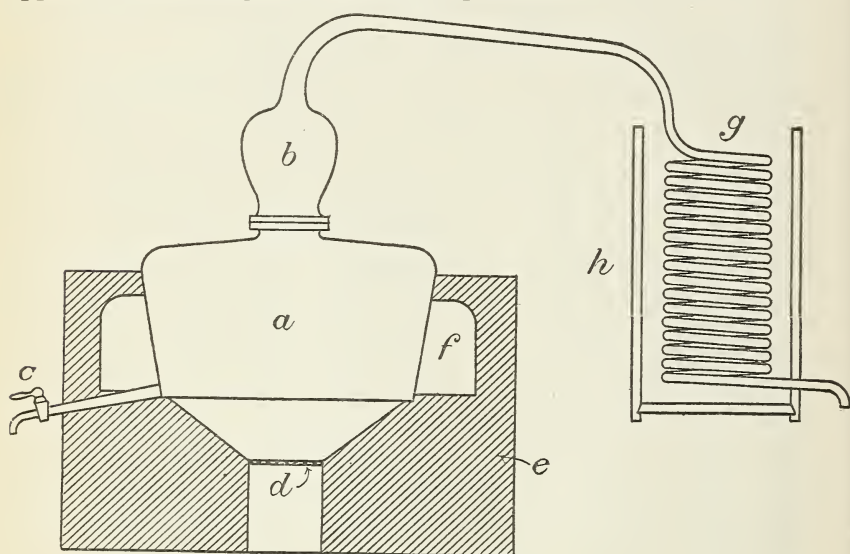
FIG. 5.—Fire pot still.

a, Kettle of still; b, head of still; c, draw-off cock; d, grate; e, masonry support; f, flue; g, condensing worm; h, worm tub.

DIMENSIONS OF STILL.

Kettle:
    50 inches diameter, bottom.
    60 inches diameter, top.
    30 inches deep to top of turn.
    Capacity, 300 gallons (full).
    Working capacity, 264 gallons (1,000 liters).
Heating surface:
    Bottom.................... 13.63 square feet.
    Sides........................ 23.12 square feet.
        Total..................... 36.75 square feet.
Grate, 15 inches square; area, 2¼ square feet.

CONDENSER.

Total surface, 35.3 square feet.
86 feet of copper pipe, 2 inches in diameter at inlet, 1 inch in diameter at outlet.
15½ complete turns, 20 inches in diameter on centers. Coils spaced at average distance of 3 inches on centers.
Tub, 30 inches inside diameter; 5-foot staves.
Capacity, 150 gallons.

When three portions of distillate have been collected, aggregating about 264 gallons (1,000 liters) at 30 per cent, the distillation of the wine is interrupted and the still is charged for the fourth run with the distillate itself, to which the name "singlings" would be given in this country.

The first portion of distillate coming over on the redistillation or "doubling" shows an initial strength of about 75 per cent and is collected until its strength is found to have fallen to about 50 per cent. A second portion is then collected, between the strengths of 50 per cent and 20 per cent, and a third portion is run from 20 per cent until alcohol ceases to come over. To these three portions the following names may be given: (1) High wines, (2) low wines, (3) tailings. The whole process of doubling will have required about eighteen hours and will have produced the following distillates:

98 gallons (370 liters) of high wines, 67 per cent by volume.
38 gallons (145 liters) of low wines, 30 per cent by volume.
22 gallons (85 liters) of tailings, 10 per cent by volume.

The high wines, or new brandy, represent the finished goods of this particular manufacturing process. The low wines are saved to be distilled with the next lot of singlings, and the tailings are added to the wine which is next distilled, so that there is neither accumulation nor loss of these weak distillates.

In running off 26,400 gallons of wine—equivalent by itself to 100 chargings of the still for single distillation—about 103 singlings will be necessary, together with 40 doublings. The product of such a season's work would be about 3,880 gallons of spirit at 67 per cent strength, equivalent to 5,200 proof gallons, together with a small residue of low wines which would be held over until another season. The necessary outlay for distillation in such a season would be about as follows:

Labor: Equivalent of 184 days of 10 hours, at $2.50		$460
Coal: 103 singlings at 210 pounds........pounds..	21,630	
40 doublings at 400 pounds........do....	16,000	
Total	37,630	
Approximately 19 tons, at $4		76
Interest and depreciation on still		100
Total cost (exclusive of water)		636

On this basis the expense for distillation alone, exclusive of water, would be approximately $12\frac{1}{4}$ cents per proof gallon, which of itself would prohibit the use of this type of apparatus in the manufacture of industrial alcohol.

The consumption of water in this still may be estimated at from 700 to 900 gallons for each run of singlings, and 1,200 to 1,500 gallons for each run of doublings, according to the initial temperature of the water and the efficiency of the condensing worm. At averages of 800 and 1,350 gallons, respectively, the whole season's work would take 136,500 gallons, or 26 gallons of water for every proof gallon of spirit production. A minimum supply of about 150 gallons per hour at a temperature not above 70° F. would be needed.

CAUSES OF UNUSUAL EXPENSE.

The expensiveness of operating the fire pot still is due to the following causes:

(1) **Imperfect utilization of fuel.**—Even though the products of combustion are made to circulate about the lateral walls of the kettle before entering the chimney they do not give up nearly so much heat as they should to the contents of the still. In such an apparatus 1 pound of coal will hardly evaporate 5 pounds of water, whereas it would do almost twice that amount of work in a good steam boiler.

(2) **Loss of time through sluggish heat transmission.**—In a fire-heated pot still the best rate of heat transmission attainable with safety is only about 2,700 B. t. u. per square foot per hour, with the entire temperature interval between the flame, or flue gases, and the liquid. In a good steam boiler a much higher rate is attainable—namely, 3,500 B. t. u. per square foot per hour.

Steam can be carried with very little loss from boiler to still and is capable of accelerating the speed of distillation very greatly. One square foot of heating surface in a properly proportioned steam coil, fed with saturated steam at 10 pounds pressure, equivalent to 239° F., will transmit 35,000–40,000 B. t. u. per hour to boiling water.

(3) **Loss of time and fuel in heating up a cold charge.**—In heating 264 gallons of fermented liquor to the boiling point, about two and one-half hours of time and about 50 pounds of coal are lost, which may be saved if the charge is heated, previous to its introduction into the still, at the expense of heat which otherwise would go to waste at some stage in the distillation.

(4) **Loss of time and fuel entailed by redistillation.**—As has been shown, about two-fifths of the labor and fuel expense of distillation is due to the need of doubling. It is plain that a great saving will result from the use of a still which will permit distillation and doubling to go on simultaneously, with only one charge for fuel and labor.

(5) **Insufficient strength after doubling.**—In the example already given, the strongest spirit produced had a strength of only 67 per cent. Under the present regulations, alcohol can not be denatured at any strength below 90 per cent; and 67 per cent goods would have to be redistilled from a pot still many times, with multiplication of expense to bring them to the higher proof.

Fortunately it is possible, by making relatively slight changes in the design of a pot still, to overcome many of the difficulties which have just been set forth, and to attain much greater efficiency. The utilization of steam for heating, preliminary warming of the liquor to be distilled, and the addition of a doubler to the still, result in much more economical production. Fig. 6 represents a still designed with these ends in view.

This apparatus differs from the simple pot still already described in having two stills placed side by side, one of which serves for the distillation of the beer or other fermented liquor, while the other is used for the simultaneous redistillation of weak distillates or "low wines." The beer in the main still is heated by means of a steam coil, and the vapors rising from this boiling beer are passed into the low wines contained in the doubler, causing them to boil in turn. As the low wines are materially richer in alcohol than the beer, so are the vapors rising from the doubler much stronger in alcohol than those passing from the still into the doubler. The pipe through which these vapors pass to the condensing worm is bent at one point into a coil of two or three turns, which is immersed in a tub called the beer heater,
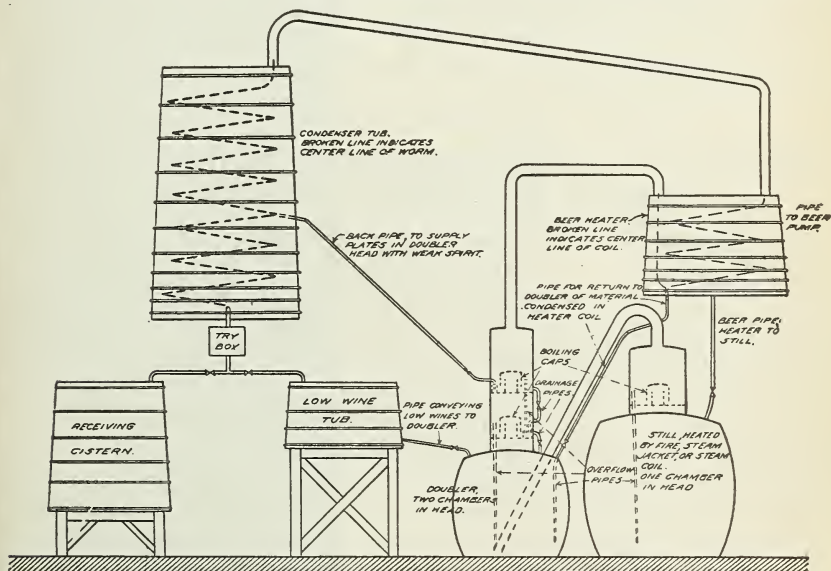
FIG. 6.—Modified copper pot still, showing doubler and connections.

capable of holding as much beer as can be distilled in one charge. Whenever the still is in operation, this tub is kept filled with beer, which is heated considerably by the hot alcoholic vapors circulating through the coil. When the beer in the still has been boiled out, this hot beer is ready for use on the next distillation. A small proportion of the vapor passing through the coil is condensed therein, and the condensate so formed is returned to the doubler by a small pipe which drains the coil at its lowest point. This condensate is weaker in alcohol than are the vapors, and therefore the coil acts as a dephlegmator.

The proof of the spirit running from the condenser of such a still is quite high at first, usually in the neighborhood of 150° to 160°, and remains high for some time, after which it falls rather slowly to

about 100°, and then more rapidly to 0°. So long as the strength of the distillate is high enough, it is run into the receiving cistern. When the proof falls below the limit which seems desirable, the distillate is turned into the low wines tub, until its proof has fallen to 0°. The low wines thus collected on each charge are reboiled in the doubler on the following charge.

In its principles, though not in form, such an apparatus foreshadows the column stills which are used everywhere for the production of high-proof alcohol. The main still corresponds to what is called the beer still in the more complicated apparatus, while the doubler corresponds to the alcohol column. The modern column stills and fractionation are discussed in the section on the experimental distillery, which follows, and in the lecture on "Effect of distillation in different types of stills" (p. 127). Various types of American stills showing in a general way the evolution of the still are pictured in fig. 7.

OPERATION OF AN EXPERIMENTAL DISTILLERY FOR MAKING INDUSTRIAL ALCOHOL.

PURPOSES AND CAPACITY OF THE DISTILLERY.

This distillery was established by the Secretary of Agriculture under the authorization of the appropriation act of May 23, 1908, with the following ends in view:

(1) To study the apparatus and processes of alcohol distillation, with the special aim of adapting the methods which have been developed in commercial distilleries in this and other countries to utilization by farming communities in such small agricultural distilleries as were authorized in section 4 of the act of March 2, 1907.

(2) To train a number of men from the various agricultural experiment stations in the application of these methods.

(3) To experiment, on a manufacturing scale, with unusual raw materials—such as farm wastes—with the aim of developing methods for their utilization in alcohol manufacture.

In order to facilitate the operation of the distillery for experimental purposes, special regulations were issued by the Commissioner of Internal Revenue, as Circular No. 721 of his office, which reduced to a minimum the amount of supervision exercised by the Treasury officials. In compliance with the provisions of this circular, the distillery was designed to have a spirit-producing capacity not exceeding 100 proof gallons daily, with the poorest material which might be employed. The daily capacity, with grain, is about 35 to 40 bushels, or 160 to 200 proof gallons.

The denaturing was done by the agent of the internal revenue according to the general formula adopted by this Government, which

CIRCULAR PERFORATIONS.

LIPPED PERFORATIONS.

SIMPLEST FORM OF

BOILING CAP.   BARBET PATENTED
                 SLIT EDGE CAP.

S OF BOILING CAPS AND
PLATES IN CONTINUOUS
BEER STILL.

HEATER

DOOJUM
PIPE.

POT STILL WITH HEATE

VAPOR PIPE TO CONDENSER

GOOSE NECK
ELEVATION

GOOSE NECK
PLAN

POT STILL WITH DOU

DOOJUM
PIPE

DOUBLER
HEAD

CHARGE
TUB.

STEAM

POT STILL;
EATED BY
TEAM SCROLL.

TANDEM STILLS A
PISTORIUS TYPE, AS U
PENNSYLVANIA RYE

SIMPLEST FORM OF POT STILL.

POT STILL WITH HEATER.
VAPOR ESCAPE PIPE.
HEATER
DOOJUM PIPE.

POT STILL WITH DOUBLER.
WORM TUB.

DOUBLER HEAD
DOOJUM PIPE
GIN HEAD, WITH PERFORATED TRAYS FOR JUNIPER.

CHARGE TUB.
DOUBLER
WORM TUB
LOW WINE PIPE.
STEAM
LOW WINE
HIGH WINE
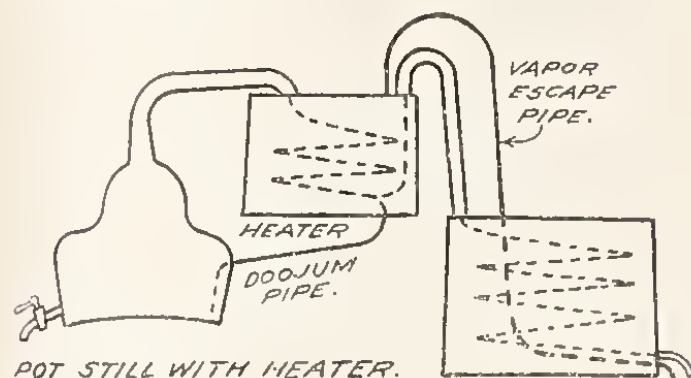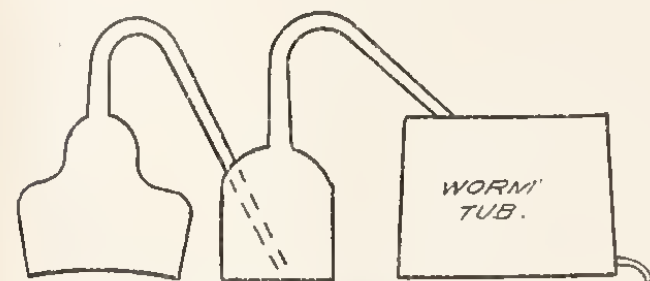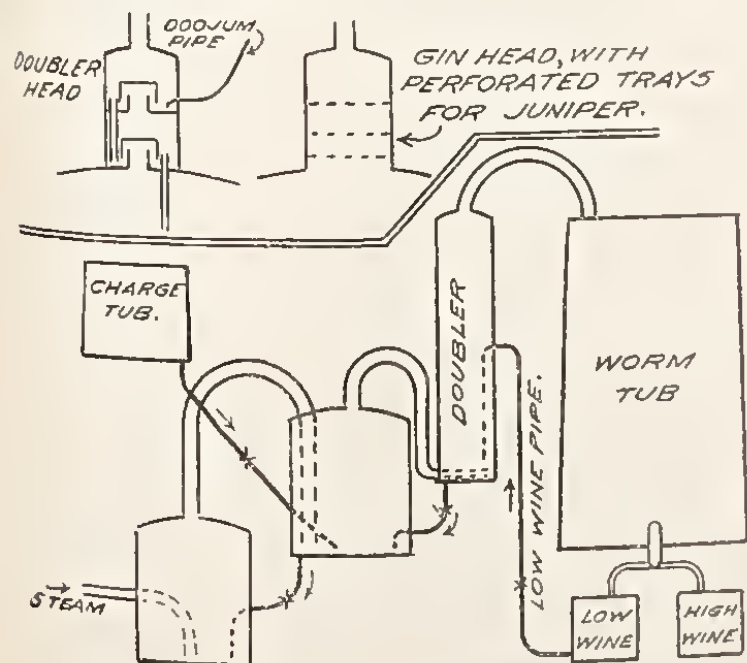
TANDEM STILLS AND DOUBLER. PISTORIUS TYPE, AS USED IN A LEADING PENNSYLVANIA RYE DISTILLERY.

CHARGE TUB.
DOUBLER
WORM TUB.
STEAM
DOOJUM PIPE
LOW WINE PIPE
LOW WINES.
HIGH WINES.
SPENT BEER
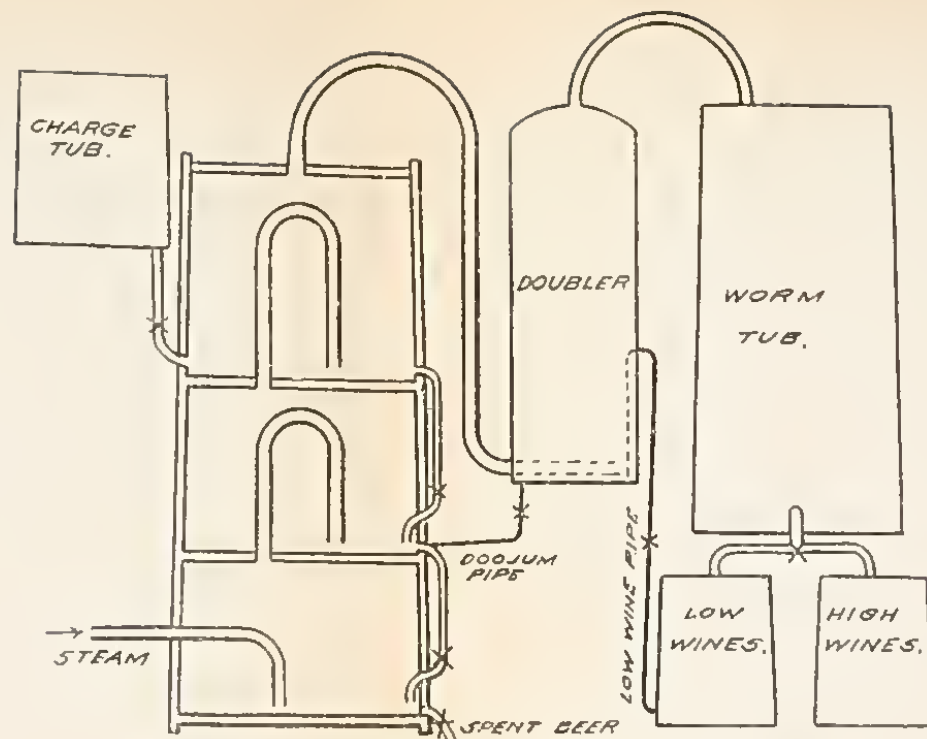
THREE CHAMBER WOOD STILL AND DOUBLER.
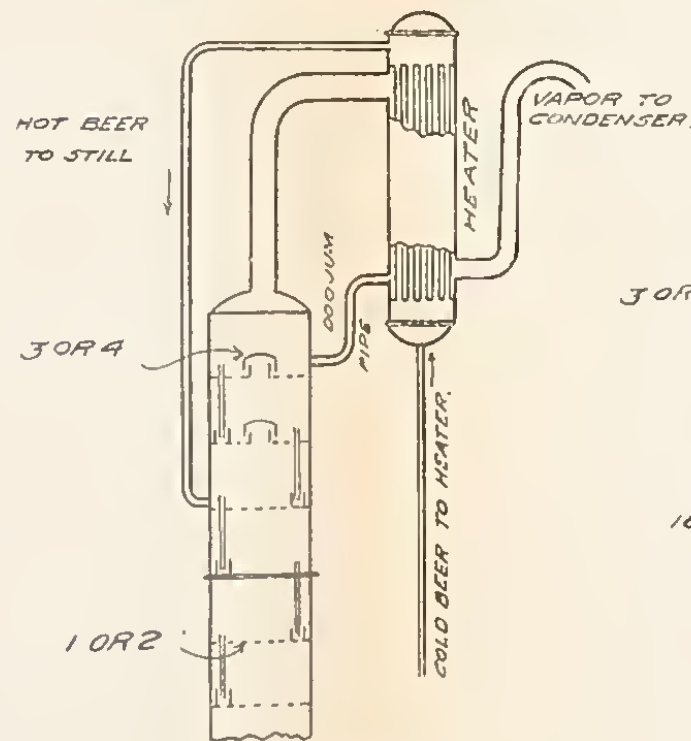
HOT BEER TO STILL
VAPOR TO CONDENSER.
HEATER
DOOJUM PIPE
COLD BEER TO HEATER.
3 OR 4
1 OR 2
FIVE CHAMBERS OMITTED
CONTINUOUS BEER STILL
STEAM
SLOP OUTLET

CIRCULAR PERFORATIONS.
LIPPED PERFORATIONS.
PLAIN BOILING CAP.
BARBET PATENTED SLIT EDGE CAP.
DETAILS OF BOILING CAPS AND BOILING PLATES IN CONTINUOUS BEER STILL.

3 OR 4
RECTIFYING COLUMN. 18 CHAMBERS.
VAPOR PIPE TO CONDENSER
GOOSE NECK.
GOOSE NECK ELEVATION
GOOSE NECK PLAN
DOOJUM PIPE
RECTIFYING SPIRIT STILL.
POT STILL; HEATED BY STEAM SCROLL.

Fig. 7.—American types of distilling apparatus.
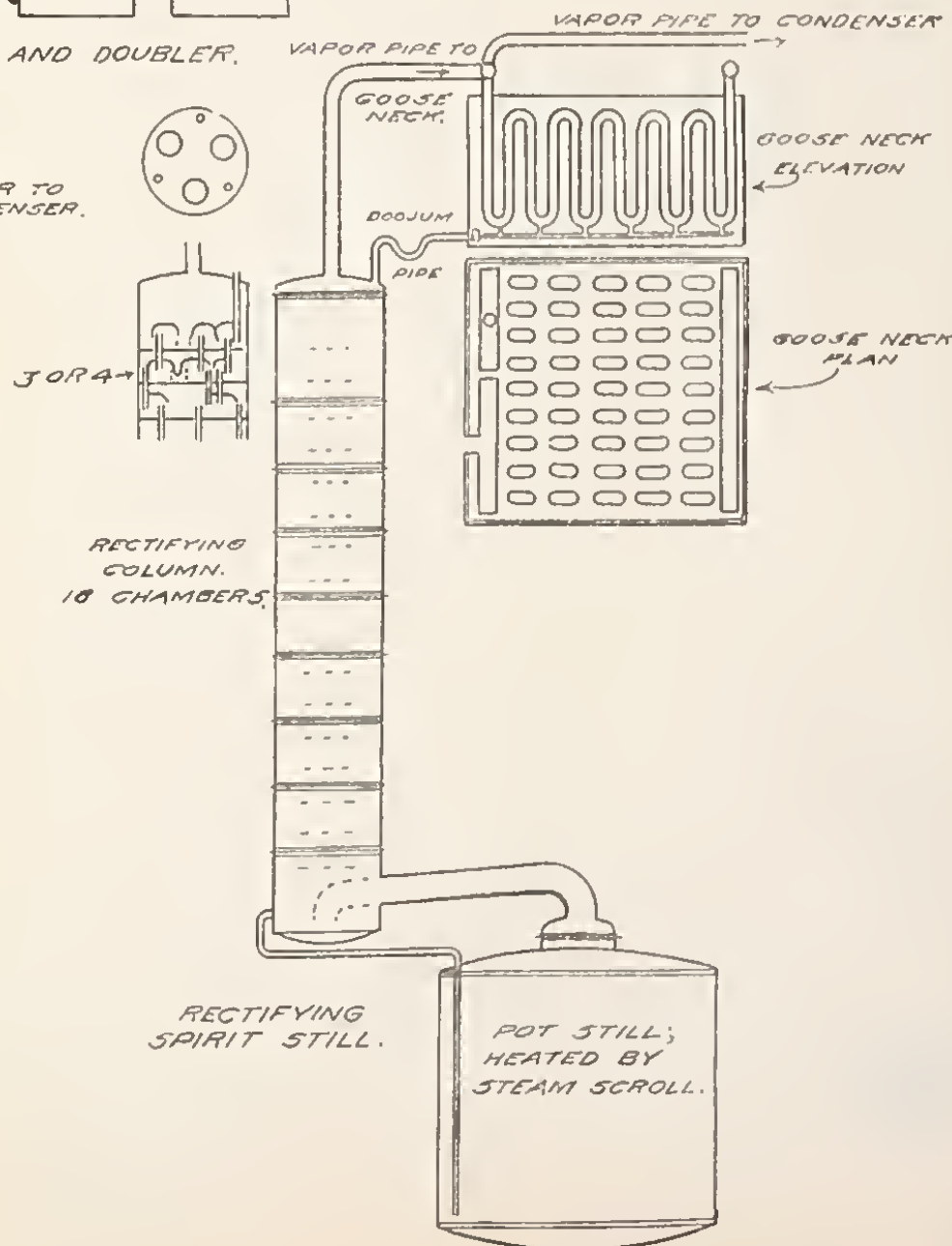
Power for the plant was supplied by a 25-horsepower electric motor, the steam being furnished by two 25-horsepower upright boilers used for heating the buildings of the Bureau of Chemistry. The plant as designed called for one 25-horsepower, horizontal, tubular, boiler which will be sufficient if the cooking and distilling operations are conducted at different times. If the two processes are carried on simultaneously, however, twice the boiler capacity will be necessary, and if steam be used instead of electricity a still greater capacity will be demanded.

It should be noted in this connection that relatively greater boiler capacity always is needed in a small distillery than in a large one. Thus, it has been found desirable in the plant here described to have a 50-horsepower boiler for mashing, pumping, and distilling, with a daily mashing capacity of about 35 bushels of grain. This corresponds to about $1\frac{1}{3}$ horsepower per bushel. On the other hand, as has been stated, a plant having a daily capacity of 1,000 bushels needs only about 600 horsepower boiler capacity, and a considerable part of this is needed only for driving the engines. These differences are due to several causes. Heat losses from radiation, etc., are relatively much greater with a small apparatus, and many heat economies which would be impracticable in a small establishment are possible in a large plant, such, for example, as the utilization of exhaust steam, of hot flue gases, and of hot water from the condensers.

COURSE OF A MASH THROUGH THE DISTILLERY.

In figs. 8 and 9 (floor plan and side elevation of the distillery) the power necessary for operating the mills and the rakes and stirrers in the cooking and mashing apparatus is generated by the motor Y. When grain is employed it is first ground in the mill V and elevated to a scale hopper on the floor above by means of the elevator U. From the scale hopper the meal is run by means of a spout directly into the cooker Q, where the mashing process is carried out as described in the section on that subject (p. 45). The small-grain tub O is used for mashing the malt preparatory to its addition to the cooked grain in the cooker. After the mashing process is complete, the cooker charge is allowed to flow by gravity into the drop tub P, whence it is pumped by means of the mash pump S through the pipe cooler M into any one of the fermenters L. While the mash is being pumped to the fermenters, the yeast, which has in the meantime been prepared in one of the two yeast tubs N, is also run into the fermenter. After the fermentation is complete, the fermented mash is dropped by gravity from the fermenters L into the beer sink K.

PE COOLER
AST TUBS
MALL GRAIN MASHER
ROP TUB
OOKER
VACUUM PUMP
ASH "
RAIN ELEVATOR
EAL "
ILL
RUSHER
RINDER
OTOR

de elevation, experimental di

A. BEER STILL
B. COLUMN
C. CONDENSER
D. TAIL BOX
E. SLOP TESTER
F. PRESSURE CAN
G. LOW WINE TANK
H. HIGH WINE TANK
I. MIXING TANK
J. BEER PUMP
K. BEER SINK
L. FERMENTERS

M. PIPE COOLER
N. YEAST TUBS
O. SMALL GRAIN MASHER
P. DROP TUB
Q. COOKER
R. VACUUM PUMP
S. MASH
T. GRAIN ELEVATOR
U. MEAL
V. MILL
W. CRUSHER
X. GRINDER
Y. MOTOR

FIG. 8.—Floor plan, experimental distillery.
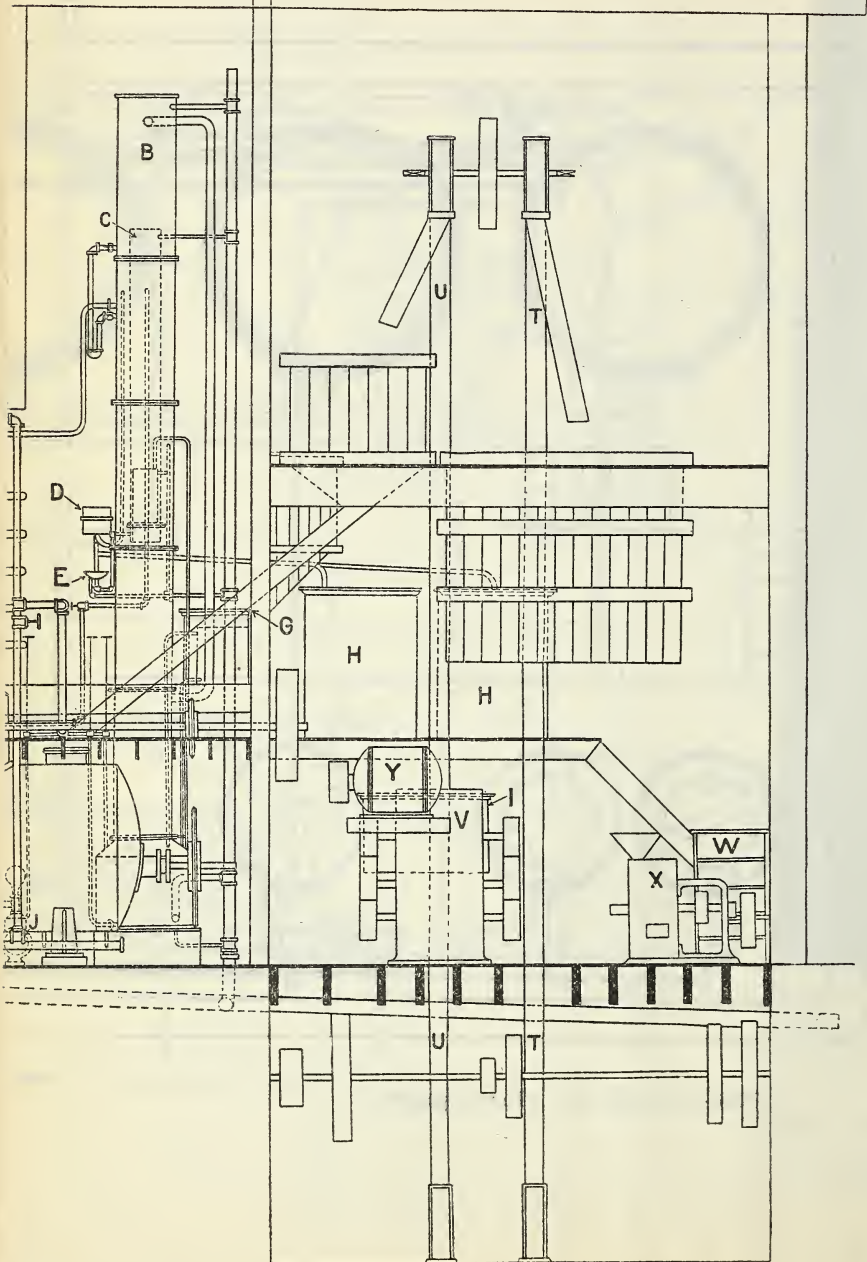
stillery.

The beer is pumped from the beer sink by means of the beer pump A (fig. 10)[a] through a pipe into the beer heater B, the course of the flow of beer being shown by the arrows. The beer passes through the heater and out at the top through a pipe, down into the top chamber of the beer still C, the pipe emptying into a cup which acts as a seal, and from this cup it flows out over the surface of the plates which form the various chambers of the still. The beer gradually flows downward over these plates, meeting a current of live steam, which enters at the bottom of the still from pipe D and passes upward through the various chambers of the still, collecting the alcohol from the beer. The beer or slop, freed from alcohol, gradually collects in the bottom chamber of the beer still, and is discharged through pipe D', which is arranged with a constant level apparatus, so that there is always a certain amount of spent slop in the chamber. The steam, enriched with the alcohol, passes up through vapor pipe E and through a tubular beer heater B, where it heats the beer and is somewhat cooled itself, so that there is a certain amount of condensation in the beer heater which is returned through pipe B' into the upper chamber of the still. The alcoholic vapor passes on from the beer heater through pipe E into the alcohol column. From the top of the bottom chamber of the beer still there is a small pipe F, which runs to a small condenser G and try-box H, where the vapors from the bottom chamber are tested to see whether they contain any alcohol. The vapors from the beer still enter the alcohol column I and pass up through the various chambers of the column, each chamber acting like a simple still, so that there are a great number of redistillations of the product before it reaches the top of the column, and thus the strength of the alcoholic vapor is greatly increased. It is in this column that the separation of the ethyl alcohol from the higher boiling-point alcohols takes place. The vapor from the alcohol column passes through the vapor pipe K into the tubular goose L. This goose is cooled with a current of water, which enters at T and passes out at U, and is so regulated as to keep the temperature in the goose at approximately the boiling point of pure ethyl alcohol, so that all substances having a high boiling point are condensed and run back into the still through the pipe S. In this way the vapor which finally leaves the goose through the vapor pipe K is kept practically at the boiling point of ethyl alcohol. These vapors pass down through the tubular condenser M and are condensed and run off into the try box N, which is supplied with a floating hydrometer that gives at all times an indication of the strength of the distillate. From the try box the condensed distillate can be run into the tank O, which is for the collection of the low wines, or it can be run through

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[a] The remainder of this description refers to fig. 10.
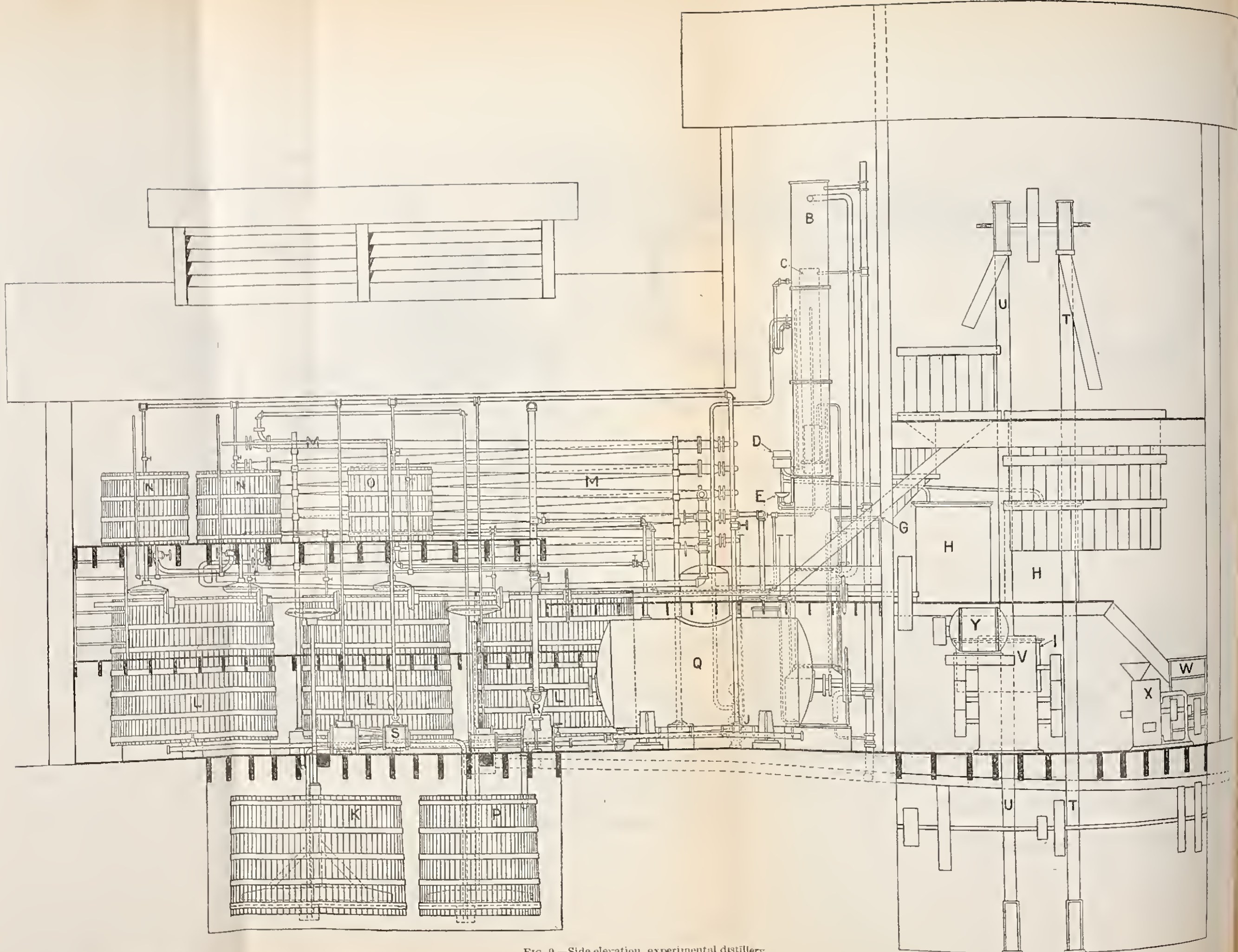
FIG. 9.—Side elevation, experimental distillery.

other pipes into the alcohol receiving tanks. The tank O is arranged so that low wines can be run back into the alcohol column for redistillation. Running from the top of the bottom chamber of the alcohol column is a small pipe P which passes through condenser G and to another try box Q. This is for the purpose of testing the vapors from the bottom of the chamber of the alcohol column so as to know whether any alcohol is left there. On the sides of the column are a number of by-passes R, which are arranged so that they can be opened to drain each one of the chambers into the chamber below. When these by-passes are opened there will be no condensed vapors in the various chambers. This still is so arranged that there is a continuous distillation of high-proof alcohol, but it will not produce so pure an article as the large column still ordinarily used in this country in producing high-proof or neutral spirits. It does, however, yield an alcohol sufficiently pure and strong for denaturing.

EXPERIMENTAL RUNS USING LOW-GRADE MATERIALS.

Corn.—The operation of the experimental distillery was begun on September 22, 1908, partly to illustrate the method of running such a still for the benefit of those attending the lecture course and also to test the possibilities of making industrial alcohol from low-grade materials. With the latter object in view, the poorest grade of corn obtainable on the market was used. Three mashes were made on successive days from this material. The plant being new and not yet in running order, great losses occurred in the handling of the first mash. A forty-eight-hour period of fermentation was used, and one of the mashes showed an attenuation of 18.5°. Distillation of the first mash was begun on September 24, the others following on the two successive days. Some trouble was experienced in keeping the waste free from alcohol and at the same time producing a spirit of 185° proof necessary to give a 90 per cent alcohol after denaturing. On the last two mashes 3 and 4 proof gallons, respectively, were obtained per bushel of corn.

During the latter part of October corn was tried again, with much better results. On October 27, 29, and 31, and on November 3, mashes were made and fermented for seventy-two hours, with an average Balling reading of 9.2° and an attenuation of 10°. The yield of alcohol ranged from 3.20 to 4.17 proof gallons per bushel of corn. Again on November 12 and 18 mashes were made from corn, and yields of 4.28 and 4.18 proof gallons of alcohol per bushel were obtained.

Watermelons.—On September 30 and on October 1 mashes were made from watermelons. The melons were very small and fully 50 per cent of them were green. They were run through the cyclone
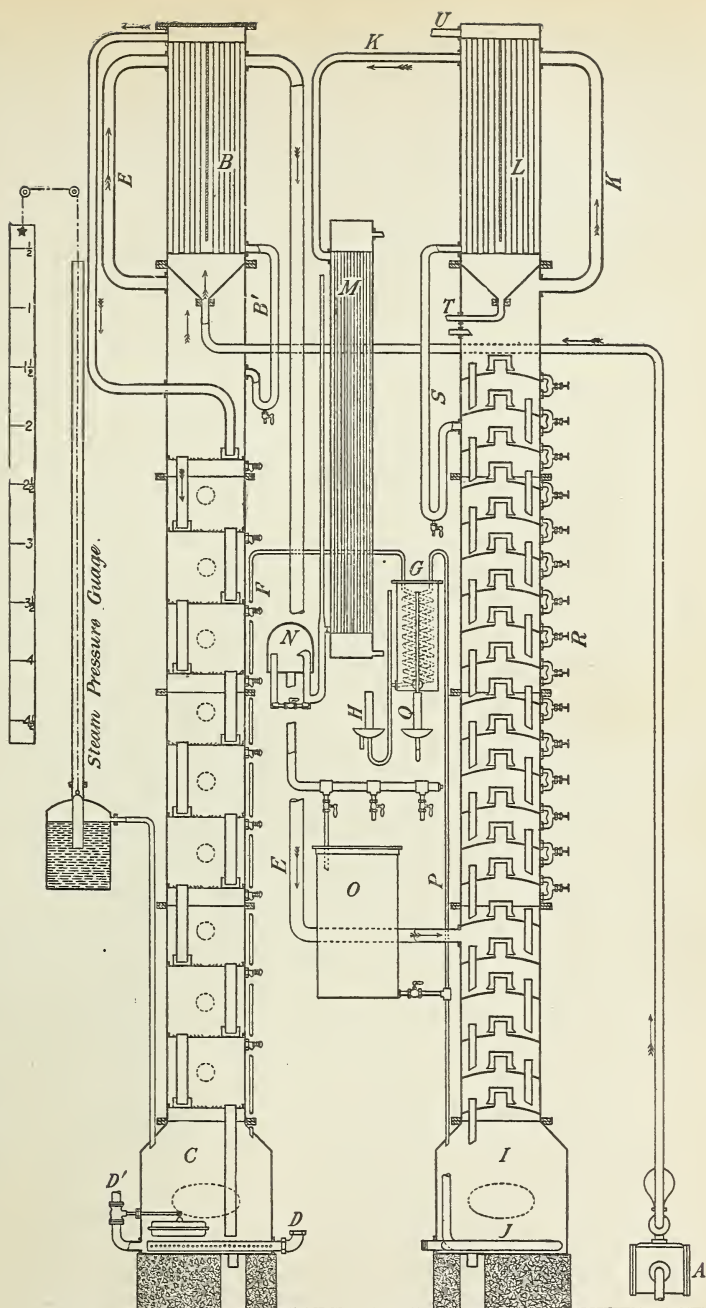
FIG. 10.—Continuous column still, experimental distillery.

A, beer pump; *B*, beer heater; *B'*, return pipe from beer heater; *C*, beer still; *D*, steam pipe; *D'* slop outlet; *E*, vapor pipe from beer still; *F*, test pipe from beer still; *G*, condenser; *H*, try box; *I*, spirit column; *J*, steam coil; *K*, vapor pipe from spirit column; *L*, tubular goose; *M*, tubular condenser; *N*, try box; *O*, low wine receiver; *P*, test pipe for spirit column; *Q*, test box; *R*, bye passes; *S*, return pipe from goose; *T*, water inlet; *U*, water outlet.

mill or thrasher cylinder and then with water through a stone mill, pumped to the cooker to be sterilized, from there to the drop tub for yeasting, and then passed to the fermenters. The mashes before fermenting contained from 3.34 to 2.69 per cent of invert sugar, respectively. Forty-eight-hour periods of fermentation were used, and an attenuation of from 3.4° to 3.7° was obtained. Considerable trouble was experienced in distillation because of the large particles of melon present, the preparation of the raw material having failed to reduce it to a sufficiently fine pulp. The yield of alcohol per ton varied from 2 to 4 proof gallons, which though small may be considered fair for that grade of material. Melons obtained earlier in the season would be much riper and their sugar content higher. Again better arrangements could be made for preparing the raw material so as not to dilute it in grinding and yet obtain a very fine pulp. In this connection the fact should be emphasized that in order to successfully utilize a variety of materials special apparatus for mashing and grinding must often be furnished, and special attention should always be paid to the appropriate preparation of the mash.

Potatoes.—On October 2 a mash was prepared from potatoes. The supply was what would be termed "offs," being very small and hardly merchantable. On analysis they yielded 15.9 per cent of starch, as compared with 18 per cent, which is considered an average amount of fermentable matter. The potatoes were run through the stone mill with water and then went to the cooker. A seventy-two-hour period of fermentation was used. On October 14 and 15 mashes from the same material were made, but with some changes in the method of preparation. In one case the potatoes were placed in the cooker without previous grinding and were treated with water and steam, in imitation of the German method. This method, while yielding starch in available form for fermentation, gave much trouble on account of the presence of peelings and uncooked lumps of potato. The fermentation of this mash was accomplished in forty-eight hours, but its distillation gave poor alcohol yields for the amount of fermentable matter present. In the next experiment the original method of preparing the mash was again used, and less trouble was experienced. The work on potatoes further emphasizes the fact that any distillery, no matter how small, must be suitably equipped for preparing the different raw materials, and in the case of potatoes the German type of pressure cooker is a necessity, the American type of vacuum cooker used in this case, while suitable for grains, having proven totally inefficient in handling potatoes.

Molasses.—On October 6, 7, 8, 14, and 15, and on November 10 and 11 mashes were made from the ordinary "blackstrap" molasses. The first two were rather heavy, the analysis showing 11.24 per cent

of invert sugar and 9.61 per cent of sucrose in one, with slightly less in the other. A forty-eight-hour fermentation period was used, and an attenuation of 14° obtained. The other mashes were made with much less molasses and averaged about 7 per cent of invert sugar and 1.58 per cent of sucrose. There was no trouble at all in controlling the yeasting, even in high degrees of concentration. The weather having turned cooler, it was possible to set the fermenter at a lower temperature and control it better during the process. The steam supply for the still became lower at this time, unfortunately, because of the greater demand of the boiler for heating the other buildings, which of course affected the distillation results, and while the yields of alcohol as figured from the beer were good, the recovery by the still was low, averaging only from 50 to 80 per cent of that obtained in the beer. This is amply accounted for by the fact that the steam supply was variable and the still was being partly run by the students attending the lecture course as a feature of their instruction.

Apples.—On November 6 and 15 mashes were made from apples. These were culls and a large part of them were rotten. The first mash was made by passing the apples with a stream of water through the cyclone cylinder and stone mills. Trouble was again experienced in getting the material fine enough and when sufficiently fine it was too much diluted. From the stone mill the material went to the cooker to be sterilized, this being necessary because of the large amount of decayed fruit present. After cooling, the material became rather thick, owing to the jelly formation of the pectin bodies. Fermentation was slow and not very complete. The second mash was obtained from the pressed juice of the apples, a cider press being used. The Balling reading at the outset was 8.2° at 79° F., and after seventy-two hours showed an attenuation of 7.3°. This mash was easily operated in the still and gave 8.4 per cent of proof spirits, equivalent to 4.67 per cent of 180° alcohol.

Partial conclusions concerning low-grade materials.—In all, 23 mashes were run, 9 of corn, 7 of cane molasses, 3 of potatoes, and 2 each of watermelons and apples. The operation of the still was not sufficiently uniform to reach any definite conclusion as to the amount of alcohol obtainable from these wastes on a commercial scale, though the yields, considering the nature of the material, were fairly satisfactory. Certain mechanical difficulties always incident to a new installation and the necessity of using the still for illustration and instruction purposes made it impossible to make record runs, but nevertheless many of the experiments gave good yields, indicating that under more favorable conditions some of these low-grade materials might prove a profitable source of industrial alcohol. Referring again to the variety of materials used, it can not be too strongly

emphasized that each material must receive special treatment in the preparation of the mash—that is, the milling machinery must be especially adapted to the different kinds of raw material. After the preparation of the mash has been successfully accomplished (that is, it is reduced to a homogeneous mass in which the largest possible amount of fermentable substance is available for the action of the yeast without undue dilution), the succeeding operations are comparatively simple and the same for all materials, with the exception that the special treatment for starch is omitted when saccharine products are used.

PHYSICAL AND CHEMICAL TESTS IN THE DISTILLERY.

SIMPLE TESTS.

The tests described in this section are so simple that they can be made by any distiller, without the assistance of a chemist. The saccharimetric determinations are made necessarily in accordance with the provisions of section 3303, Revised Statutes, and the others are so instructive that they should always be made if possible.

It will be observed that all temperatures are given in Centigrade in this section, and the same statement holds true of the work on enzyms and indeed of nearly all of the strictly scientific control work, while the distillery and operation data in general are given in Fahrenheit. This confusion is regrettable, but it did not seem wise to attempt uniformity, inasmuch as the trade and commercial usage is in fact to use Fahrenheit, and the Centigrade readings to convey their meaning must be converted. By the following formulas the readings may be readily changed one into the other: $°F. - 32° × 5/9 = °C.$ $°C. × 9/5 + 32 = °F.$

SACCHARIMETRIC DETERMINATIONS.

Two of the most important determinations in distillery practice, namely, the saccharine content of the mash and the apparent attenuation of the beer, are made by means of the saccharimeter. This instrument is nothing more than an ordinary specific gravity spindle, but so calibrated as to read directly on the scale in per cent of sugar. The Balling saccharimeter, as almost universally used in distillery work, reads at 20° C. directly to tenth per cents of solid matter in solution. The most convenient form of this spindle is provided with a thermometer and table of temperature corrections in the stem.

In determining the saccharine content of a mash by means of the saccharimeter, there are a number of points to be taken into consideration. It must be remembered that the mash filtrate contains not only fermentable sugars but also a considerable amount of nonfermentable organic and inorganic substances, all of which are indicated as sugar.

It is not customary to apply a correction for the soluble nonsaccharine matter in the mashes. The apparent saccharine content is usually all that is determined; it should be remembered in this con-

nection that with a given material of a given quality the relation of saccharine to nonsaccharine matter is nearly constant. In determining the attenuation of a beer it is the apparent attenuation and not the actual attenuation which is ordinarily determined. This apparent attenuation is of course greatly affected by the amount of alcohol produced during the fermentation, but for a given material and concentration of the original mash it is a direct indication of the completeness of the fermentation. One degree of apparent attenuation is equivalent to about $0.8°$ actual attenuation and indicates in general the conversion of 0.8 per cent of sugar, referred to the amount of beer, into about 0.5 volume per cent of alcohol.

COMPLETENESS OF SACCHARIFICATION OF THE MASH.

For this examination 5 grams of iodin should be dissolved in a small amount of water containing 10 grams of potassium iodid, and the resulting solution diluted to 1 liter. This solution should be prepared afresh at frequent intervals, as the color reactions for which it is used are not always reliable when produced by a solution which has been kept in stock in the laboratory for a considerable period of time.

The test must be made in an absolutely clear filtrate from the mash to be examined. About 10 cc. of the clear filtrate is brought into a small porcelain dish and 1 cc. of the iodin solution added. The color produced will range from deep blue through violet and red to colorless. The blue color is produced by starch, the violet by an intermediate product between starch and dextrin, the red by dextrin. The malto-dextrin and maltose produce no coloration. A faint reddish color is permissible in a satisfactorily saccharified mash.

ACIDITY DETERMINATIONS.

The determination of acidity in distillery control is usually made by direct titration of a measured volume of clear filtrate against standard sodium hydroxid solution. Acidity is seldom expressed in percentage, but ordinarily in degrees, and it is unfortunate that no standard value for a degree of acidity exists. Where acidity is mentioned in this report it is determined as follows:

Twenty cc. of the clear filtrate is titrated against normal sodium hydroxid, and the number of cubic centimeters of the latter used in the titration represents the acidity. If it were found, for example, that 3.1 cc. of normal alkali were necessary to neutralize 20 cc. of filtrate, then the acidity would be expressed as $3.1°$.

It is sometimes desirable to distinguish between fixed acids and volatile acids. The following method is by no means exact, but the results obtained are of sufficient accuracy to be of value in distillery control.

Determination of volatile acids (butyric, acetic, etc.).—One hundred cubic centimeters of clear filtrate are concentrated by vigorous boiling in a porcelain dish over a direct flame to about 20 cc. and then cooled and diluted to its original volume. The fixed acidity is determined in 20 cc., as previously described, and the difference between this result and the total acidity is considered to represent the volatile acids.

DIASTASE IN THE FERMENTED MASH.

Dissolve about as much as a pea of guaiacum resin in 2 cc. of alcohol in a test tube. Add to this about 10 cc. of filtrate from the fermented mash. If precipitation occurs add a little more alcohol. Now add about 1 cc. of hydrogen peroxid. If diastase is present a blue coloration will take place immediately or within a few moments. The rate at which the color forms and the intensity of the color give a measure of the quantity of diastase present. This reaction is probably not due to diastase directly, but to the presence of certain oxydases which always occur with it and the destruction of which would indicate the destruction of the diastase. When negative results are obtained by this method it is advisable to make a blank test, using the same reagent mixture and a few drops of malt extract.

The presence of diastase may also be ascertained by the use of a little soluble starch. A few drops of a 2 per cent soluble starch solution are placed in a test tube, about 10 cc. of clear filtrate from the fermented mash added, and the mixture kept at a temperature of 60° C. for a period of about one hour. It should be tested from time to time with a little iodin solution. The disappearance of the blue starch coloration indicates the presence of diastase, and the rate at which it disappears is indicative to some extent of the quantity of diastase present.

MOISTURE IN GRAIN AND MALTS.

Moisture in malt should always be determined by the official method described on page 38, in Bulletin No. 107, Revised, of the Bureau of Chemistry. It may be determined in grain in the same way, but much time may be saved by employing a method devised in 1905, in the Bureau of Plant Industry.[a] The fundamental principle of the latter method "consists in heating whole grains in oil to a temperature considerably above that of boiling water, and thus driving out the water, which is afterwards condensed and measured in a graduated flask. With this method it is possible to determine the percentage of water in a sample of corn in from twenty to twenty-five minutes." The method reads as follows:

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[a] A quick method for the determination of moisture in grain. By Brown and Duvel. Bul. 99, Bureau of Plant Industry, U. S. Dept. Agr. This publication may be obtained from the Superintendent of Documents, Government Printing Office, Washington, D. C., price 5 cents.

One hundred cubic centimeters of a good grade of hydrocarbon oil are measured and poured into a glass distillation flask [fig. 11]. One hundred grams of corn are weighed on a torsion balance * * *, the corn being emptied at once into the flask containing the oil. The neck of the flask is closed with a good rubber stopper carrying a thermometer, the bulb of which should extend well into the mixture of oil and corn. The neck of the flask is then connected with a condenser by means of a second rubber stopper. With a strong gas burner the corn in the oil bath is then heated until the thermometer registers 190° C., at which time the flame is extinguished. The time required for the temperature to reach 190° C. will be from ten to fifteen minutes, depending on the amount of water in the corn and on the volume of the flame. Eight or ten minutes after the flame has been removed the water will have ceased dropping from the condenser, and the number of cubic centimeters of water contained in the graduated cylinder beneath the condenser tube can be ascertained. This is the water actually removed from the corn and represents the percentage of water originally in the sample, each cubic centimeter of water representing 1 per cent when a 100-gram sample is used.

This method is not suited to use with malt, as the results are likely to run high.

ANALYTICAL DETERMINATIONS.

Unlike the preceding tests, the following determinations call for a well-equipped laboratory and the services of a chemist. On this account they will hardly be adopted as part of an established distillery routine, save in large plants. At times, however, emergencies may arise in small plants which will necessitate the making of such analyses. The order in which the methods are described indicates in a general way their relative importance.

FIG. 11.—End view of apparatus for moisture determination in grain.

A, condenser; *B*, evaporating chamber; *C*, stand supporting condenser and evaporating chamber; *c*, overflow pipe; *d*, washer soldered to bottom of condenser tank; *e*, rubber stopper supporting condenser tube; *g*, screw bolts; *i*, ledge for supporting gauze, triangle, and flask; *i'*, galvanized iron ring above gauze; *j*, flanged pipestem triangle; *k*, covers; *m*, handle on cover; *p*, distillation flask; *q*, thermometer; *r*, rubber stopper; *s*, condenser tube; *t*, graduated measuring cylinder; *v*, braces; *x*, burner; *x'*, extra stopcock beneath burner; *y*, gas pipe; *y'*, support for gas pipe.

DIASTASIC POWER OF MALT (INCLUDING PREPARATION OF SOLUBLE STARCH).

This determination is fully described in the lecture on chemical control of distillery operations (page 117). The method which is recommended therein for recording the diastasic power of a malt

(ratio of the amount of sugar formed in a standard time, to the amount of malt employed) is far better suited to distillery work than the usual system, which involves the use of arbitrary degrees.

STARCH AND SUGAR IN RAW MATERIALS.

For these determinations use the methods of the Association of Official Agricultural Chemists, as given in Bulletin 107, Revised, Bureau of Chemistry, under the following headings: For starch, see page 53, section (b), Diastase method with subsequent acid hydrolysis; for sugar, page 64, under Methods for the analysis of saccharine products, or page 79, under Fruits and fruit products.

PROTEIN IN RAW MATERIAL AND SLOP.

Use methods given in Bulletin 107, Revised, page 38, under analysis of foods and feeding stuffs.

UNGELATINIZED STARCH IN THE MASH.

In cases of poor fermentation in which the fresh mash has shown evidence of unusual amount of starch, a quantitative determination of the ungelatinized starch is often of value.

Weigh out 1 kg. of the thoroughly mixed mash and dilute with about 10 liters of water in a tall glass bottle. Allow to settle for twenty-four hours and siphon off the supernatant liquid. Mix the residue thoroughly with a second 10 liters of water and again allow to settle. This second settling will usually require from six to eight hours. Siphon off the supernatant liquid and repeat the process until the insoluble residue has been washed in this manner ten times. Bring the residue onto a filter and wash with alcohol and ether. Dry thoroughly in the air and weigh; use 3 grams of this dried residue for the starch determination, as previously given.

FERMENTABLE SUBSTANCES IN THE FERMENTED MASH.

Three hundred cubic centimeters of clear filtrate from the fermented mash are so far neutralized with sodium hydrate that it still shows 0.3° acidity. It is then concentrated on the water bath to about one-third volume to remove the alcohol which it contains, cooled, diluted to its original volume, and treated as follows:

(a) One hundred cc. are set with 2 grams of pure compressed yeast and allowed to stand at about 30° C. for twenty-four hours. It is then brought into a suitable distilling flask with 100 cc. of water, and 100 cc. distilled off and carefully condensed. Alcohol is determined in this distillate by means of a pycnometer.

(b) One hundred cc. are subjected to the action of 10 cc. of malt extract and then treated exactly as under paragraph a.

(c) One hundred cc. of the malt extract are fermented and the alcohol determined exactly as under paragraph a.

One-tenth of the alcohol found under paragraph c is subtracted from the alcohol found under paragraph b, and the difference between the result and the amount of alcohol found under paragraph a is considered to represent an alcohol loss due to imperfect saccharification.

Fermented mash residues showing by the above treatment a yield of alcohol of less than 0.3 per cent may be considered as satisfactorily fermented.

ALCOHOL IN THE FERMENTED MASH AND SLOP.

In distillery control the determination of alcohol is ordinarily made on the fermented mash before distillation and very frequently on the slop during the distilling process. It is an all-important point that the sample used for the determination of alcohol should be a representative one, and due care must be exercised in procuring it. In the case of very thick mashes it is difficult to distil off the alcohol from a small quantity in the laboratory without burning the grains. But with sufficient care the distillation may be effected successfully and the use of corrections for volume, which are apt to introduce considerable errors when large factors are concerned, may be avoided.

From 250 to 500 cc. of the beer or mash is placed in a suitable flask connected with an ordinary condenser and the alcohol distilled off and collected in a suitable graduated flask of from 100 to 300 cc. capacity. The distillate is made up exactly to the mark on the flask and its specific gravity very accurately determined by means of the pycnometer. From a table of specific gravities with corresponding alcohol strengths of mixtures of alcohol and water the actual amount of alcohol in the sample under examination may readily be found.

A further discussion of this determination is given in the lecture on the chemical control of distillery operations on page 121.

DENATURED ALCOHOL AND DENATURANTS.

By C. A. CRAMPTON, *Chief Chemist, Internal-Revenue Service.*

DISCUSSION OF TERM " DENATURED ALCOHOL."

The term "denatured alcohol" was new to most persons prior to the passage of the law. The noun was well known, of course, but the qualifying adjective was peculiar and its significance was not generally understood.

About the time the bill first went into effect a letter was received in the correspondence of the internal-revenue office from the high-school principal of an eastern city, asking what was meant by "denatured alcohol," and stating (what was doubtless the truth at that time) that little or nothing could be found in the dictionaries or cyclopedias concerning it. Even the general public, however, has become pretty well instructed in the significance of the term since the law went into effect, and most persons understand that denatured alcohol is alcohol to which some substance or substances have been added which render it unpotable, thus making it unfit for use as a beverage, but not for industrial purposes. It might be considered as an equivalent of the term "dehorned" applied to cattle; that is to say, "denatured alcohol" is alcohol deprived of its power for evil and then let loose from government surveillance and control for purposes of peaceful and useful employment.

The word is not quite so descriptive as one could wish; it seems to imply that something is taken from the alcohol, whereas something is always added to it; moreover, the essential nature of the alcohol is not changed. However, it was early chosen as being the best that offered; the word "depotabilized," while more descriptive, being too ponderous for ordinary use, and "denaturized" or "denaturalized" having the same fault, although preferable etymologically. "Industrial" alcohol is a good term to describe the purpose for which it is intended, but does not necessarily imply denaturation.

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DENATURING SYSTEMS AND FORMULAS OF DIFFERENT NATIONS.

It is doubtless known by most of the audience that the United States was about the last of the leading countries of the world to provide for the use of alcohol free of tax in the arts and industries. Most of the earlier bills proposed for the free use of alcohol were based upon a rebate system of control, the alcohol to be used, tax-paid, in some line of manufacture, and the tax then refunded upon proper proof being made of its having been used as prescribed. This system implies a very rigid and constant supervision over factories where alcohol is used and a close accounting for raw materials and finished products. It is unsatisfactory from the government point of view, because of the difficulty in proving fraud and the time that must elapse between the perpetration and the discovery of the fraud, and it is unsatisfactory to the manufacturer because of the long delay to which he is subjected in the slow process of refunding. It could only be applied, of course, to alcohol used for manufacturing purposes.

The law finally adopted, however, relies mainly upon what might be called a chemical control of the tax-free use of industrial alcohol, the alcohol being removed from bond, without payment of tax, as soon as it has been mixed with a suitable denaturing material. Most of the European nations adopted systems of this kind during the second half of the last century, that of England dating back to 1855. These systems are all very similar in principle, though they differ greatly in detail. Most of them provide for two classes of denatured alcohol, viz, that known as "completely," "absolutely" or "generally" denatured alcohol, intended to be sold at retail to the public for the general purposes of heat, light, power, and cleaning; and that known as "incompletely," "specially," "relatively," or "partially" denatured alcohol, intended for use in lines of manufacture where the substances added to completely denatured alcohol would militate against its use, or where the use of the alcohol under bond justifies a denaturant which is not so fully protective in quantity or character. For complete denaturing methyl or wood alcohol is almost universally used, denatured alcohol in England being still popularly known as "methylated spirits," the name that was originally applied to it when wood alcohol was the sole denaturant. It was found in time, however, that the wood alcohol alone was not a sufficient addition to prevent the drinking of methylated spirits, and it was supplemented with a small quantity of mineral naphtha, or kerosene, and the alcohol thus denatured, properly but not generally

designated as "mineralized methylated spirits," is the kind sold in England at the present day for heat, light, and cleaning.

The merits of wood alcohol as a denaturant, therefore, were early recognized, and nothing has been found to supplant it, as it forms a part of every formula prescribed in Europe for complete denaturing. The specifications as to the character of the wood alcohol vary in different countries, but all prescribe a certain amount of impurity. This partially refined product is pungent and repellent, and it is impossible to separate it from ethyl alcohol except by the elaborate and costly processes of fractional distillation. It is easily recognized in extreme dilution by delicate chemical tests, and constitutes, therefore, a means by which the use of renatured alcohol in drinks can be discovered, as even fractional distillation will hardly remove it entirely so that its presence can not be demonstrated. Moreover, it has good solvent powers, burns as readily as ethyl alcohol, and does not greatly exceed it in cost, so that a reasonable quantity of it added to the latter does not detract from the uses for which the denatured alcohol is intended or add materially to its cost. Its use alone, however, is not sufficient to fully safeguard from drinking the alcohol with which it is mixed, as has been shown by the experience of England, just cited. It must be supplemented, therefore, with some other repellent. In the case of England, as already stated, this addition is a small amount of kerosene, English completely denatured alcohol or mineralized methylated spirits containing 9 per cent of approved wood alcohol and 0.375 per cent, or three-eighths of 1 per cent, of mineral naphtha (kerosene). The French formula is very similar to the English, except that 10 volumes of approved wood alcohol are added to 100 volumes of ethyl alcohol, making the mixture contain slightly over 9 per cent, and the wood alcohol is supplemented with 0.5 per cent of a heavy benzine, a coal-tar benzine of a fixed type. The function of the hydrocarbon in both cases is to form a milky emulsion when the alcohol is diluted to beverage strength, and thus make it repugnant alike to sight, smell, and taste.

The German completely denatured alcohol is probably not so well protected against renaturing as the English-French, as it contains only 2 per cent of approved wood alcohol, and the difficulties of separation and of its detection by the methyl reaction in drinks wherein it is used are correspondingly diminished. On the other hand, it is probably more repugnant to the sense of smell and taste, as the wood alcohol is supplemented with 0.5 per cent of pyridin bases, very bad smelling substances, formerly obtained by the distillation of bones or other animal substances, but now mainly derived from the volatile by-products of the distillation of coke in closed retorts. It is permitted to the denaturer to soften the rigors of this perfume by the

further addition of 0.125 per cent of rosemary or lavender oil, but this privilege is not often utilized. The odor of pyridin is not only very disagreeable and lasting, but it has a very decided physiological effect when inhaled, producing headache and nausea. I must also mention the formula for the completely denatured alcohol sold in Germany for use in internal-combustion engines. This contains 1 per cent of wood alcohol, 0.25 per cent of pyridin bases, from 2 to 20 per cent of benzol, and a small amount of methyl violet dye.

The formula formerly used in Austria-Hungary was the same as the German, with the further addition of a trace of phenolphthalein, a substance which is colorless in acid solution, but bright pink in alkaline solution, this addition being evidently made to provide a ready and simple means for distinguishing between ordinary alcohol and denatured alcohol. The Austrian formula was modified about a year ago, by the introduction of benzol, and an additional substance, the nature of which is kept secret. In Switzerland a somewhat complicated formula is used, and it is changed from time to time by the Government without notice. In Belgium no provision is made for complete denaturing, the general sale of alcohol for popular purposes not being permitted, although it can be used tax free in the manufactures, for which special denaturants are provided.

Notwithstanding the fact that the formulas for complete denaturing are not entirely satisfactory in fulfilling their functions, so that the search for better materials for serving the purpose is constantly going on (both France and Russia having offered prizes for more satisfactory formulas), no radical changes have been made in them for many years. The difficulties in the path of improvement in this direction are well shown by the following specifications, imposed by the French committee on analytical methods, upon the competitors for the prize offered by the French Government, in connection with the law of November 29, 1905:

This act instituted two prizes: One of £800 for the discovery of a denaturing agent more advantageous than those now used, while safeguarding the revenue against frauds, and a second (£2,000) prize for a system of utilizing alcohol for lighting in the same manner as paraffin. The denaturant must have a taste and smell which will effectually discourage any desire to use the alcohol as a beverage. The denaturant should not be sufficiently objectionable in smell to prevent its domestic or industrial use. No soluble substance which could leave a deposit on lampwicks, and thus render combustion difficult, may be used. It must not consist of a substance much more or less volatile than alcohol and which could thus be removed by fractional distillation. It should contain no substance which might injure the metallic part of lamps or motors. It should not be poisonous or contain poison. It should be sufficiently inexpensive, should not normally exist in commercial alcohol, and its presence in alcohol should be capable of easy and certain detection.[a]

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[a] J. Soc. Chem. Ind., 1907, *26*: 994.

DENATURING FORMULAS ADOPTED BY THE UNITED STATES.

After examining the different formulas used for complete denaturing in Europe, witnessing the manner of their application, and consulting the officials charged with the chemical control of the use of tax-free alcohol, I had no hesitation in recommending to the Commissioner of Internal Revenue the adoption of the English-French rather than the German-Austrian formula, as the former is more efficient in preventing drinking and renaturing, while at the same time it is not so disagreeable to use. Wood alcohol is less expensive to produce here than in Europe, and the formula should be a very cheap one and add little to the cost of the alcohol. This recommendation was adopted and the regulations so issued. Shortly before the law took effect, however, it appeared that certain interests were able to control the production of wood alcohol of a grade suitable for denaturing purposes, and intended to maintain a high price for it outside of their own distilleries. Therefore, the Commissioner of Internal Revenue asked for a formula for complete denaturing which would not require the use of wood alcohol at all. This I was unable to furnish, but suggested that the German formula be authorized as an alternative, thus cutting down the amount of wood alcohol required to less than a fourth of that used in the formula already put out. This was done, and the action dispelled whatever danger there had been of a monopoly of wood alcohol, which went to a parity of price with denatured alcohol on the day the law took effect, and has maintained about the same relation ever since.

It is therefore optional with the distiller in the United States to use either of the two generally adopted formulas in the complete denaturing of alcohol, a degree of liberality which does not obtain in any other country, although it is paralleled to a slight extent by the French provision for the sale of "finish" and the German provision for the sale of alcohol denatured specially for use in engines, both in addition to the sale of alcohol completely denatured according to the usual formula. As a matter of fact, little or no advantage has been taken here, so far, of the alternative formula, practically all completely denatured alcohol being denatured according to the English-French formula. It was tried in Milwaukee, where there is a large number of citizens of German nativity, but even there it met with disfavor on account of its ill-smelling character.

The specifications imposed upon the materials submitted for approval for use in completely denatured alcohol will be found on pages 22–26 of Internal Revenue Regulation No. 30. In the case of wood alcohol maximum and minimum limits are imposed upon the acetone and the bromin absorbing constituents, and a maximum limit upon the

esters. The requirements are more elastic than those imposed in Europe. The "benzine" may be derived either from coal tar or petroleum, but the specific gravity and boiling-point requirements insure a heavier product than that which is popularly known as "benzine" in this country, and this has led to some misapprehension about the character of the benzine of the regulations. Ordinary kerosene answers all the requirements, and is the material which has been used, without exception, in all the completely denatured alcohol marketed so far. A cheaper kerosene, or one not too highly refined, is more apt to meet the requirement as to rendering the alcohol unfit for use as a beverage.

The specifications imposed upon pyridin bases follow German precedent, and the small quantity used in this country so far (chiefly in special denaturants, the German formula for complete denaturing having found no favor here, as already mentioned) has been imported from Germany. It is produced here in considerable quantities, however, or rather could be produced if it were saved as a by-product from the purification of ammonia obtained from the condensation of the gases from coke ovens. At present it is allowed to go to waste in the air, the demand for it not justifying the cost of a plant for saving and purifying it, according to the statement of large producers of alkali by the ammonia-soda process.

SPECIAL DENATURANTS.

Coming now to the subject of special denaturants, it will be found that the systems employed in different countries are essentially similar though differing greatly in details. A manufacturer of a product made from alcohol, or in the manufacture of which alcohol is used, gives bond that he will use the alcohol only for the purpose specified, and his entire premises are subject to the inspection of revenue officials and liable to forfeiture if any improper use is discovered. In consideration of the additional security thus afforded, he is permitted to use denaturing materials which are better adapted to his line of manufacture, even though such materials do not protect the alcohol against misuse as well as do the materials added to completely denatured alcohol. It is to the interest of the manufacturer himself that some repellent substance be added, in order to protect him against his workmen, who might otherwise drink or carry off the alcohol. The purposes for which alcohol is permitted to be used entirely without addition are very few, indeed, in any country, and our law makes the addition of some material "which destroys its character as a beverage and renders it unfit for liquid medicinal purposes" a necessary antecedent to the withdrawal of alcohol from tax liability. Germany

allows the use of pure alcohol, free of tax, in all public hospitals and institutions of learning, and for the manufacture of munitions of war. We have practically the same concessions in our statutes, enacted prior to the denatured alcohol law, which permit the use of tax-free alcohol for scientific and governmental purposes.

Some industries are very difficult to provide with suitable denaturants, on account of the nature of the products or the character of the processes employed; others are quite easy, the materials added in the usual course of manufacture effectually destroying the alcohol or rendering it undrinkable. This latter is known as "ad hoc denaturing," where an agent is used that is a necessary part of the process of manufacture instead of being a substance entirely foreign to it. For example, sulphuric acid added to alcohol in the manufacture of sulphuric ether, and castor oil and soda in the manufacture of transparent soap, are ad hoc denaturants. This kind of denaturing has the merit of economy, of course, as the denaturing materials serve a double purpose, and do not increase the cost of the alcohol; but it is not always applicable, and frequently the use of a small quantity of a substance foreign to the process, but highly repellent, like pyridin or animal oil, answers the requirements better than the ad hoc denaturant. The ad hoc system is quite highly developed in France. The authorized formula there for use in the manufacture of chloroform, for instance, is 5 kilos of chlorid of lime to 100 gallons of alcohol; for the manufacture of ethyl chlorid, equal parts of hydrochloric acid and alcohol; for the manufacture of chloral hydrate, "a current of chlorin gas is passed through the alcohol," and so on. Thus the process of manufacture itself often serves the purpose of insuring the proper use of the alcohol, and the officers are only required to witness the initial step, which consists of the addition of the strong chemicals. In some cases the application of a definite degree of heat is prescribed, as a part of the denaturing operation, to insure the beginning of the chemical reaction.

The German system of special denaturing is more liberal than that of any other European country; the required additions being often very small in quantity and of slight deterrent efficiency or easily removable. Another liberal feature is the wide range of choice in formulas permitted to manufacturers.

In England manufacturers were not allowed the use of special denaturants up to the time of the passage of the law of 1902; they were obliged to make use under bond of methylated spirits, which were denatured with 10 per cent of wood naphtha. The law passed in 1902, however, was very liberal in its provisions, even allowing the use of pure spirits without addition, if necessary. The whole matter of determining what materials could be used, however, was placed in

the hands of the officials of the board of inland revenue, who could use their discretion in granting suitable denaturants, and they have been very conservative in this direction. They do not publish any set list of authorized special denaturing formulas, and seem to make it a matter of direct arrangement with individual manufacturers. They state in their annual report for the year ended March 31, 1907, as follows: "There is good reason to believe that the industrial purposes are relatively few of which the requirements in respect of alcohol can not be satisfactorily met by the use of 'industrial methylated spirits' [since the law of 1906 denatured by the addition of 5 instead of 10 per cent of wood alcohol, practically equivalent to our formula No. 1], and in future it is probable that the cases which will require to be dealt with under section 8 of the finance act, 1902, will not be numerous."

Germany, however, allows the use in any line of manufacture on bonded premises of alcohol denatured with 2 per cent of wood alcohol alone, or 0.5 per cent of pyridin alone—the two ingredients of the complete denaturing formula—and provides also an extensive list of the special denaturants for optional use. It must be kept in mind that the conditions in Germany are quite different from those in England, as the higher tax on distilled spirits in the last-named country offers a much greater inducement to fraud; and in this respect our own conditions approximate more closely those of England than those of Germany.

Under our law there have been authorized to date seventeen different formulas for special denaturants for use in the various lines of industry in which alcohol plays a part. They will be found listed in Supplement No. 1[a] of the regulations. The aim is to list the lines of manufacture according to the final product as produced and sold, but a rigid classification is difficult to follow because of the diversity of ways in which alcohol is employed in the arts and industries. Formula No. 1—the addition of 5 gallons of approved wood alcohol to 100 gallons of ethyl alcohol—is used far more than any other formula, being authorized for use in more than twenty-five different lines of manufacture. Alternative formulas are authorized for twelve different lines; that is, either one of two different formulas may be used. The following is a list of the various substances used as denaturing materials, some of which enter into several different formulas: Wood alcohol (two different grades), camphor, benzol, castor oil, caustic soda, coloring material, sulphuric ether, cadmium iodid, ammonium iodid, pyridin bases, condensed fulminate fumes, acetone, petroleum naphtha, zinc chlorid, sulphuric acid, nicotine, kerosene, and animal oil.

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[a] See page 161 of the Appendix.

A list of the various industries in which the use of special denaturing formulas have been authorized will be found in Supplement No. 1 of the regulations.[a]

The conditions under which special denaturants are authorized for use are set forth in section 6, Part VI, of the regulations. Manufacturers are presumed to be in a position to determine what agents can be used in connection with alcohol without detriment to their product. Formulas are submitted to the Commissioner of Internal Revenue, who passes upon their sufficiency, and determines whether they should be authorized. Experience has shown that manufacturers are apt to act too hastily in applying for formulas before making experiments to determine whether they could be used; in several cases formulas have been authorized upon the application of manufacturers, who subsequently found that they could not be used, and were obliged to make application for changes, thus delaying the utilization of tax-free alcohol in their line of industry. Concerted action on the part of manufacturers in the same line of industry is greatly to be desired; some manufacturers seem to get the idea that if they can hit upon a satisfactory denaturant and secure its official authorization they should obtain a reward for their enterprise in the shape of a monopoly of the use of alcohol in their line—a sort of a patent, as it were. This can not be allowed, of course, as the law must be executed to the benefit of all persons alike.

CONCLUSION.

As this address treats the subject in only a very general way, all members of this audience who are chemists and wish to make a closer study of the subject of existing systems of denaturing and of denaturing materials are referred to an English official publication, the Report of the Departmental Committee on Industrial Alcohol, published in 1905 by Wyman & Sons, Fetter Lane, London, E. C. This contains an account of the system of denaturing employed in each country, together with all formulas for special denaturing, and much other interesting matter in this connection, making it the most comprehensive publication on the subject up to the time of its issue. An excellent resumé of it, which is readily available to chemists, may be found in the issue of the Journal of the Society of Chemical Industry for April, 1905, volume 24, pages 397 to 426.

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[a] See page 164 of the Appendix.

MISCELLANEOUS LECTURES.

Forty-two lectures were delivered during the alcohol course, a list of which follows. Those given by members of the Bureau of Chemistry, especially when pertaining to the operation and description of apparatus as illustrated by the experimental still, are not given in lecture form, having been for the most part incorporated in the preceding sections of the report, in which it has been endeavored to give as a consecutive narrative the fermentation and distillation processes relating to the production of industrial alcohol, and thus avoid duplication. A few were not reported at all, as the exhibits shown in connection with them were necessary to their presentation. Other lectures, dealing with the possible sources of industrial alcohol, the laws and regulations regarding the same, details of operation as set forth by practical distillers, etc., are given substantially in full, even at the risk of a little repetition, inasmuch as they answer in a simpler and more popular manner than the more extended treatise many questions constantly asked at the Department. References are given in the list to the page where the lecture, or a digest thereof, may be found.

PROGRAMME FOR COURSE ON DENATURED ALCOHOL.

October 5 to November 10, 1908.

Purpose and scope of proposed course on fermentology (see page 86). By H. W. Wiley.

Machinery for grinding and operation. By A. Given.

Molasses as a source of alcohol. By H. E. Sawyer.

Beet molasses. By A. H. Bryan.

Mashing machinery. By A. Given.

Description of still. By L. M. Tolman.

Cassava as a source of alcohol (see page 99). By C. C. Moore.

The chemistry of barley and malt (see page 36). By J. A. Le Clerc.

Potatoes as a source of alcohol (see page 97). By H. W. Wiley.

Mashing: Principles of operation. By H. W. Berger.

Cactus as a source of alcohol (see page 105). By W. W. Skinner.

Prickly pear and its fruits as a source of alcohol (see page 107). By R. F. Hare.

Bananas as a source of alcohol (see page 104). By G. N. Collins.

Yeast—pure culture preparation. By H. W. Berger.

Cereals as a source of alcohol (see page 94). By H. W. Wiley.

Manufacture of baking yeast. By H. W. Berger.

Principles and theory of fermentation.   By H. E. Sawyer.
Control of distillery (see page 117).   By A. M. Breckler.
Yeast and yeasting (see page 122).   By A. M. Breckler.
Distillery economics.   By A. M. Breckler.
Practice of fermentation with cane molasses.   By H. E. Sawyer.
Bacteriological technique (see page 133).   By G. W. Stiles.
Use of alcohol in explosives and plastics.   By C. E. Munroe.
Distillery practice (see page 109).   By J. A. Wathen.
Yeasting (see page 113).   By J. A. Wathen.
Yeasts for fruit fermentation.   By W. B. Alwood.
Yeast culture.   By W. B. Alwood.
Microscopical control (see page 136).   By B. J. Howard.
Distillery practice (see page 125).   By R. P. O'Brien.
History of distillation (see page 90).   By H. W. Wiley.
Practice in the use of potatoes, beets, and molasses.   By H. W. Berger.
Fractional distillation in different types of stills (see page 127).   By L. M. Tolman.
Chemistry of alcohols and their derivatives—aldehydes, acids, esters, etc.   By
H. W. Wiley.
Utilization of denatured alcohol for power (see page 141).   By R. N. Strong.
Regulations (see page 152).   By Wales Hubbard.
Denaturants and denaturing (see page 76).   By C. A. Crampton.
Denatured alcohol (see page 146).   By J. G. Capers.
Uses of denatured alcohol in medicine.   By L. F. Kebler.
Uses of denatured alcohol in varnish making (see page 138).   By F. P. Veitch.
Closing remarks on the denatured alcohol course (see page 157).   By H. W. Wiley.

The following lecture, delivered as introductory to the course, explains the purpose and scope of the "school on fermentology."

About 75 attended the opening lecture, the attendance often reaching 100 and over during the course and including representatives from 8 experiment stations, chemists from the Internal-Revenue Service and the Department of Agriculture, and several distillery chemists, some of whom contributed valuable lectures on the practical operation of a still, necessary machinery, etc. The experiment station representation was as follows: L. B. Broughton, Maryland Station; H. D. Spears, Kentucky Station; G. J. Morton, North Dakota Station; J. A. Hummel, Minnesota Station; R. F. Hare, New Mexico Station; C. A. Cathcart, New Jersey Station; J. W. Ames, Ohio Station; and C. E. Bradley, Oregon Station. The Georgia Fruit Association was represented by E. F. Kitchen.

PURPOSES AND SCOPE OF PROPOSED COURSE ON FERMENTOLOGY.[a]

By H. W. WILEY, *Bureau of Chemistry.*

For two years a statute of the United States has permitted the withdrawal of alcohol for technical purposes free of tax, provided that such alcohol be so denatured that it can be used for no other purpose than that intended. One of the purposes of this act was to encourage the manufacture of alcohol on a small scale by farmers or associations of farmers. It is realized that in the manufacture of alcohol on a large scale,

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[a] Introductory lecture, delivered on October 5, 1908.

where, for instance, thousands of bushels of grain are utilized each day, the economies of the process are such that the product can be made at less expense, gallon for gallon, than when made on a small scale. For instance, it would be quite impossible, in so far as economy of supervision and labor are concerned, to manufacture alcohol in a still producing 100 proof gallons per day in competition with a still producing 20,000 proof gallons per day. It is evident, therefore, that if the manufacture of alcohol in a small way is to become successful it must be due to other advantages than those accruing from economy of manufacture. These advantages it is thought are found in the fact that on farms, especially large farms, are often found great quantities of fermentable matter representing a waste product which could be utilized for the manufacture of alcohol at practically no expense for the raw material. In an instance of this kind it would be possible for the small factory to make a product which could be used in competition with that made by the large factory. Thus the farmer growing potatoes, sugar beets, Indian corn and other cereals, fruits, watermelons, etc., might utilize the waste products of his crop, or those parts of it which can not be profitably used for other purposes, in the manufacture of alcohol. Again, it is found in many instances that the cereal crops especially are often injured by frost or wet weather to such a degree that the product is not suitable for food either for man or for other animals. It is well known that certain fungus diseases so affect cereal crops at times as to render them injurious when fed to cattle, and a definite disease known as pellagra is probably due to the utilization of musty Indian corn for food purposes. Many hundreds of thousands of bushels of such injured grain are constantly produced, and although it would not produce so great a quantity of alcohol as the sound grain, yet its utilization in this way means practically its saving for an economic purpose. Growers of watermelons, of fruits, etc., also often leave upon the ground quantities of material containing notable percentages of fermentable matter which are left to waste. The canners of sweet corn produce yearly many thousands of tons of waste material particularly rich in fermentable matter. Since it has been demonstrated by the Bureau of Chemistry that the cobs and refuse coming from such canneries are particularly rich in starch and sugar and yield a high percentage of alcohol, it seems advisable to study this problem of the utilization of farm wastes of a fermentable character to the fullest extent possible, and at the same time to devise apparatus of moderate capacity which would be suitable for operation either by farmers, or, better, by syndicates of farmers.

Although the law relating to denatured alcohol has been in force now for two years, no attempt has been made, so far as is known, by any farmer or syndicate of farmers or other persons or corporations to manufacture alcohol on a small scale from such waste material. In order to secure the full benefits of the law along these lines, the Secretary of Agriculture recommended to Congress the appropriation of a small sum of money for the purpose of establishing an experimental distillery, representatives of the agricultural experiment stations of each State to be invited to participate in the study of its practical operation. To this end there was erected on the premises of the Bureau of Chemistry a small distillery with a license capacity of 100 proof gallons of alcohol per day, to be operated under the auspices of an official connected with the Department of Agriculture and to be utilized as a training school for the purpose of instructing young men in the theory and art of distillation, in order that similar establishments may be erected in the different States. This course of instruction marks a new era in the efforts of the Department of Agriculture to devise methods of giving knowledge relating to agriculture in harmony with the act passed in 1862 establishing the Department. That act does not limit the methods by which knowledge may be disseminated, and it is undoubtedly a proper function of this Department to organize courses of instruction in agricultural sciences similar to that which we are beginning to-day. It may even be that this beginning marks an era in the establishment at Washington of what may be called a graduate school in agricultural science which

will in future years become practically a great university. It is recognized that agriculture is the basic industry of the United States and will continue to be such no matter how rapidly the manufacturing and transporting industries may be developed. Not only are we called upon to feed and clothe our own people, but to contribute largely to the supplies of other nations. Hence it is eminently proper that every product of agriculture be utilized in the most economical way. It has been shown in other industries that the utilization of the waste products often marks the difference between success and failure. May we not safely predict that the agriculture of the future may depend largely upon the proper utilization of its waste products? The present hour, therefore, may be put down as the beginning of this new era of instruction of a post-graduate character in agricultural technics.

A word now in regard to the experiments which have been proposed for this course of instruction. In planning a new departure of this kind it is quite impossible that the method should be the most suitable one that could possibly be devised. It must be at best a tentative plan, and as outlined at present may undergo certain changes during the course of instruction. It is evident from the replies received from the agricultural experiment stations and others who have been invited to send students to this school that there will be two classes of students present—those who have come, as you have, at the beginning with the expectation of remaining until the close of the course, and those who come in for a week or ten days and who will receive instruction only in regard to some special phases of the investigation. At best the latter can only learn the technical part of the process and that largely by observation. On the contrary, those attending the whole course will cover the technical part of the process not only by observation but also by actual operation of the fermentation and distillation processes; they will also receive a course of lectures, about forty in number, on the theory and practice of fermentation and distillation, and practical instruction in the application of denatured alcohol for various purposes. In other words, the object of the study is that you should be able after its completion to supervise the erection of a distillery and conduct it successfully, thus fulfilling the principal purpose for which this course of instruction is established, namely, to secure in as many States as possible a center of instruction from which the knowledge of the proper method of making denatured alcohol may be given to the farmers of the State.

The lectures will consider, first, all the various kinds of raw materials which may be utilized for alcohol making and their relative importance. It is assumed that you are all familiar with the ordinary processes of analysis, so that very little time need be given to the methods of ascertaining the quantity of fermentable matter which may be found in each kind of raw material, and, as it is important to know what are the principal sources which promise most for this purpose, particular attention will be devoted to the wastes of the farm—injured grain, unmarketable melons, refuse potatoes, fruits, and other products of a similar character. Those who are near large cities will also be instructed in the wastes of the market, which are extremely valuable as a rule for alcohol-making purposes. For instance, in the case of sweet Indian corn alone there are in large cities hundreds of tons of such material which are either kept so long that they are no longer suitable for consumption or have grown too hard or too soft for edible purposes. All this material, including the cobs, is most valuable for making alcohol. You will be instructed next in the methods of grinding these raw materials so as to prepare them for the next step, which technically is known as mashing—that is, heating to a high temperature for the purpose of breaking the starch granules and preparing them for the hydrolysis. The next process is that of malting, and the purpose of malting and the method of conducting the operation in the mash tubs will be considered. Following this comes a study of the preparation of the yeasts of various kinds, the temperature at which they are

most effective, and the general technic of fermentation. This will be naturally followed by a study of the different forms of stills and the purpose for which the modifications of stills are made and the technical process of the still, both in the separation of the alcohol, first from the beer, and second in the concentration of that alcohol to a degree which renders it suitable for technical purposes.

Following the instruction on the process of distilling, comes a study of the process which is described by the Bureau of Internal Revenue for denaturing a product. The process is divided into two parts: First, the denaturing for ordinary purposes, such as fuel and light; and, second, denaturing for special purposes in manufacturing. All of these processes will be duly illustrated. A final step in the course will be instruction in the theory and practice of applying denatured alcohol, both for manufacturing purposes and for heating, lighting, and the driving of machinery. In so far as possible practicable demonstrations of these various purposes will be made, that you may become somewhat acquainted with the art as well as the theory of this application.

Another purpose which will be served by this course of instruction will be the correction of many errors known at present respecting the manufacture of denatured alcohol. Many farmers have believed that the production of alcohol is a simple matter which any unskilled person may undertake. This course will illustrate how erroneous such a notion is. Alcohol can only be produced by those who are skilled in the art, and it is not in the nature of things to expect that its production will become general, even on large farms. The best that can be expected is that the manufacture of alcohol will be conducted by cooperative establishments as at present is the case in the thrashing of wheat or the milling thereof. Much disappointment has naturally followed the enactment of this law by reason of the fact that denatured alcohol is at present made only by the large distilleries and at prices which are prohibitive as compared with those of gasoline. This is only to be expected when we consider the price which at present must be paid for the materials used in fermentation. With Indian corn well above 50 cents a bushel in price, and being regarded the cheapest and at present the only source of alcohol, it is unreasonable to expect that the price of the article shall be very low. A bushel of corn will make a little over 2 gallons of denatured alcohol, and thus at the start it is seen that the price of the raw material is well up to, if not above, 25 cents. Add that to the cost of manufacture and it is obvious that the actual cost of a gallon of denatured alcohol at the present time must be well above 30 cents, probably 35 cents. Add now the profits which the manufacturer or transfer company and the retail dealer are reasonably entitled to receive and it is no wonder that it is offered at retail at from 45 to 60 cents per gallon. If all these facts were known, the erroneous notions relating to this business might be removed. Another point which must also be borne in mind, however, is that the supplies of gasoline and other fuels are limited in so far as is known and we are drawing upon stores which will not be replenished. On the other hand, the art of agriculture will continue, and hence it is reasonable to expect that the price of denatured alcohol will fall and the price of gasoline will rise. Also every one will prefer denatured alcohol because of its greater safety and less unpleasant odor. In order, therefore, to develop this industry as rapidly as possible some such course as this is necessary. While this instruction may also be obtained at technical schools, these are more local in character and their advantages less readily available. What is offered here is free to every State in the Union which sees fit to take advantage of the investigations we are making. I hope that your stay here may be pleasant and profitable and that you may carry back to your station that knowledge of the theory and art of distilling which will enable you to begin similar instruction at home.

THE HISTORY OF DISTILLATION. [a]

By H. W. WILEY, *Bureau of Chemistry.*

The works on the history of chemistry and alchemy give little information respecting the history of distillation, and articles in the encyclopedias and dictionaries furnish but brief outlines on the subject. A few years ago distillation was generally thought to be an art which was not much practiced before the ninth or tenth centuries, at which time it was known in Arabia, and was carried by the Arabians to various parts of Asia and Europe. Later researches seem to indicate that the art is much older than that, and distinct references are made to it in the literature of events almost ante-dating historic times. Documents have been found which carry the date of this art back to 800 B. C. Distilled liquor was known as "arrack" by most of the eastern nations, and the Chinese, Japanese, and Indians have names for it almost alike in sound, which have been known from the earliest historic times.

The word "chemistry" is supposed to be derived from one of the old names of Egypt, which was "cham," "chemi," or "chemia," as the art of chemistry was first practiced in that country. The French for water bath is "bain marie," which is derived from one of the early Egyptian chemists, a woman named "Mary." The art was practiced in Egypt long before the Christian era. The word "alcohol" is supposed to be of Arabian derivation, "cohol" meaning any finely powdered or pure substance, and the word "alcohol" in later works on pharmacology means any finely divided article. For instance, a finely powdered substance such as flour would have been called "alcoholized." So any process by which the finer parts are separated from the grosser parts would produce a substance known as "cohol," the syllable "al" being also of Arabic origin, as in the word "alembic." Thus the term "alcohol" referred not only to distilled spirits but to all finely divided materials as separated from the grosser parts; just as the word "distil" did not mean originally the separation of the vapor from a liquid or solid, but the separation of the finer parts from the grosser parts. The Latin "distillo," from "de stillo" (from stills) means a drop, and the process of obtaining this liquor was called distillation. So the term "alcohol" still retains something of its early significance.

The art of distilling wines is evidently of French origin, although the distilling of water and nitric acid and many other kinds of distillation were practiced long before the French, apparently about the eighth century, began to separate the volatile substance from the wine, and called it "eau-de-vie," which is the common name in French. "Eau-de-vie" is derived solely from wine, and is known in this country as brandy, a word that the French do not use. "Brand" means "burnt" wine. Any wine sent to be distilled would be set aside to be burnt, because fire was used in separating the volatile portions. Hence the term "brandy" is simply derived from the old French word "brandevin," and from the German "branntwein" also.

The art of distilling volatile substances, or the fermented mash of grain, was probably first practiced with rice. It was done at a very early date in the extreme south of England, in Cornwall, and in Ireland along about the year 1000 or 1100. When Henry II visited Ireland he found that the distillation of grain was practiced in that country. So the art of making whisky, or the distilling of grain, is older or about as old as that of distilling wine in France. It is supposed by some historians that the art of distilling in Ireland was known long before this period, so it is probable that the distilling of alcohol from fermented grain may be perhaps older than the Christian era in Ireland and England.

Thus it is seen that distillation is a very old practice, and while its early history is deficient, the references to stills and distillation in early literature, even by Pliny, show that they were known to exist. Records show how sailors obtained drinking water by holding wet cloths over boiling sea water and then squeezing out the condensed water. This is one of the earliest known condensers. When a liquid is boiled in a covered vessel, before the lid gets hot we obtain condensed water from the under surface of the cover. This was undoubtedly the first observation of distillation, and it is exactly the same principle that is employed in distillation to-day.

To study the later evolution of this art is more important, however, than to discover its very early history, and we have in the books of the alchemists descriptions and numerous sketches of various forms of stills which were used. In the early times the stills were made in the shape of animals (fig. 12). One of the earliest forms of stills of which we have a record is shown as *a* in fig. 13. You see how small it is, and
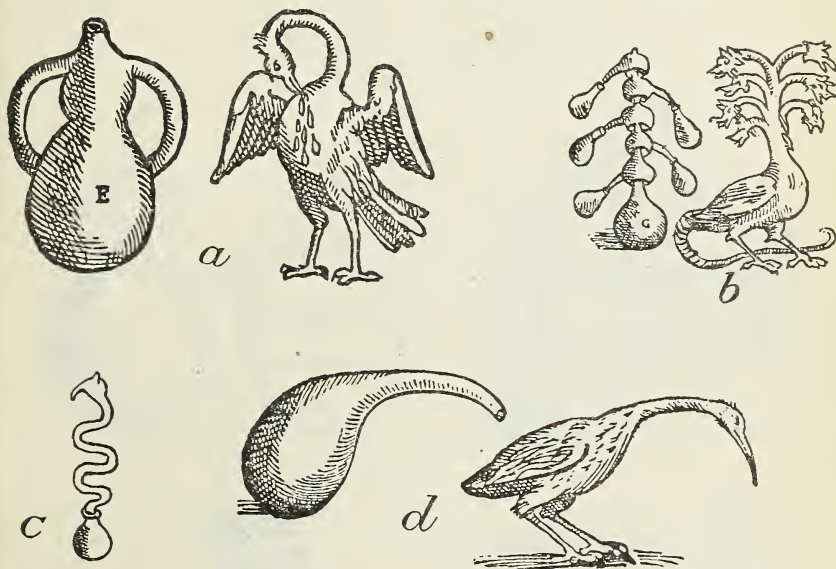
FIG. 12.—Early stills in the shapes of animals (Fairley).
a, Pelicanum; *b*, Hydra; *c*, Serpentino; *d*, Ciconia.

yet it embodies every element which goes to make up the modern still. This is said to have been copied from the ancient Temple of Memphis, in Egypt, so this form is many hundreds of years old. Cut *b*, one of the ancient forms of stills, is used to-day in China. The distillate is collected in the vessels shown. Fig. *c* is a still which was found in use in India. Cut *d* is a kind of still used by the savages in the Society Islands. The heads are put on by clay, and the small tube in which the distillate is condensed is shown and also the receiver. Still *e* was found in the ruins in Peru. The tube of this still is concave; the pan at the top is filled with cold water, and the hole closed with clay—a very ingenious and instructive form of still.

In the earliest practice, no attempt was made to increase either the purity or the strength of the distillate by means of fractionating devices in the same still; but, however, many of the alchemists thought that if you distilled five times, you secured the best distillate. Hence, this was called "quintessence." This repeated redistillation was the only means of increasing the strength or purity of the distillate. In modern times the still has been changed from the original forms, with the idea of increasing the strength and purity of the distillate by fractionation.

This development of modern times can be shown by the changes which have taken place in the manufacture of stills. In the Scotch whisky distillery the boiling heads of the still have been greatly elongated, and in many cases have a bend in the vapor pipe cooled by water to condense the heavier boiling portions and to give a higher proof, making, for all practical purposes, a redistillation still similar in its effect to
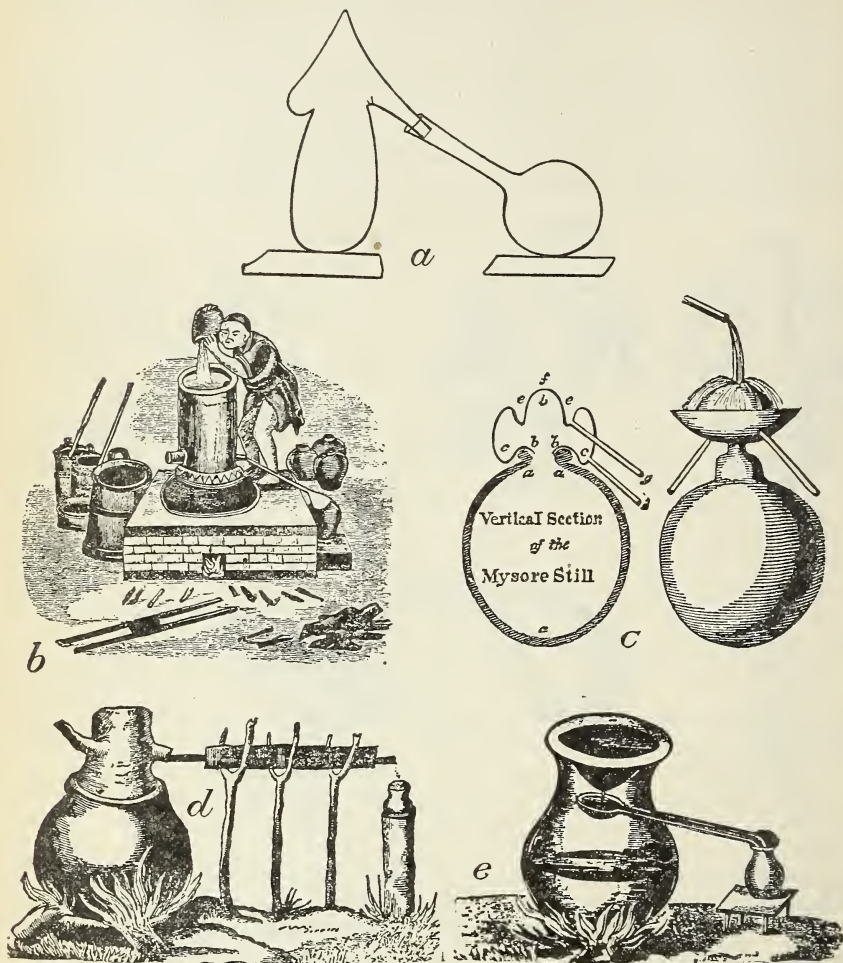
FIG. 13.—Some ancient forms of stills (Fairley).

a, Alembic and receiver from Temple of Memphis, Egypt; *b*, still used in China; *c*, ancient still used in southern India; *d*, primitive still used in the Society Islands; *e*, ancient form of still used by Peruvians.

the small doubler now ordinarily attached to most of the stills in use in this country. From this single pot still with a bend in the vapor pipe, it may be said, has developed the two-chambered still, as exactly the same principles are involved, the vapor from the first still entering the second still at the bottom and boiling the liquid which has condensed. The two-chambered still was naturally followed by the three-chambered still; this by the three-chambered still with a small doubler attachment, which is for all practical purposes another chamber; and from this three-chambered still has come the continuous still originally known as the Coffey still of England,

which was apparently the first of this type. The development of these stills is shown very satisfactorily in fig. 7, giving the outline drawings of stills as they have gradually developed from the single chamber into the many-chambered continuous still and the rectifying column used to-day in producing high-proof alcohol. The Coffey still consists of two columns filled with a long series of chambers. The material to be distilled enters at the top of one of these columns, and gradually works its way to the bottom, meeting a current of live steam coming in the opposite direction, which removes the alcohol and carries it off through a vapor pipe into the second column, where it is fractionated as it works up from the bottom of the column to the top of a complex series of plates.

In this country the continuous beer still of the same general type as the still in use in the Department distillery is the final development of the modern still as far as simple distillation is concerned. The question, however, of fractionation, as developed in the modern column used in an alcohol distillery, is not along the line of the modification of simple stills, but is based on a different consideration; that is, a development of the fractionating tubes which have been used in chemical analysis for a long time. The still used in the Department distillery is very closely related to the original type of the Coffey still, and is not used to any extent in this country in the production of pure high-proof alcohol, which is nearly always accomplished in a still separate from the beer still. This is done in order that certain processes of purification, such as filtration through charcoal and chemical treatment, may be applied before the final distillation, and also because the kettle still with a column upon it is much more easily regulated than a still of the type used here, and also it is possible to separate the distillate into various proportions according to their purity, resulting in a much purer product than that obtained from a continuous still in which there is no appreciable difference in the purity of the distillate at any time.

The possibility of producing a pure alcohol by the column still, as used in this country, can be readily seen when the practice in vogue in these distilleries is considered. After the product from the beer still has been diluted to 100° proof, it is filtered through charcoal to remove the odor and taste, and then treated with such chemical reagents as may be necessary to remove impurities, as acids, esters, and aldehydes. It is then placed in the large kettle. When the distillate begins to come over, the first portion, say 5 per cent of the total quantity, is cut off and set aside and called "head" alcohol. Then again, as the distillation proceeds, it is the practice of certain distilleries to make several fractions of varying purity. As the distillation approaches the end a certain proportion of the tail alcohol is cut off, which will contain the larger part of the impurities. This head alcohol and tail alcohol constitute the crude commercial product used for ordinary purposes, and for burning, and is available for the making of denatured alcohol.

The pure spirit obtained in this kind of a still is the very highest grade that can be made by distillation, and is known to the trade as cologne, German, French, neutral, or silent spirits, and comprises about 85 to 90 per cent of the whole distillate, the other 10 per cent going into the heads and tails. As the distillation goes on, there is a continuous separation of the higher alcohol from the ethyl alcohol, and this higher alcohol gradually accumulates on the plates, working its way to the bottom of the column. As the alcohol becomes exhausted or is driven out of the kettle below, the liquid in the lower chamber gradually becomes turbid, and there is a separation of these higher boiling point alcohols. This turbidity and separation of the higher alcohols gradually works from the bottom chamber toward the top chamber, and when practically all of the ethyl alcohol has been driven over, most of the fusel oil and higher boiling point alcohols will be collected in the upper chambers of this column; when distilled, what is known as the fusel oil of commerce is obtained.

It is rather strange that until fifty years ago the pure spirits which were used in the arts were made by chemical means. The making of pure spirits mechanically, as it is

now conducted, was not understood, and the chemicals combined with the impurities either destroyed them or held them in combination, so that afterwards the alcohol could be improved by filtering through charcoal. None of the products made in this way were so pure as those made by scientific fractionation. If you can get a constant boiling point you can get a chemical unit. The spirit made by fractionation is far more pure than any chemist can make by chemical treatment, and, more than that, the chemical treatment is so expensive and the product would be so high in price that it could not compete with the fractionated product in the markets. And so the old methods of rectification by chemicals are not used.

Theoretically, to determine the specific gravity of alcohol, the water is removed by chemical means and the result is an absolutely pure alcohol. I doubt if this has ever actually been done, but the chemist gets what is practically pure alcohol, only a very minute quantity of water remaining. The idea that alcohol is solely a product of fermentation is not correct. Like many other products—sugar, for instance—it has been made synthetically, but the cost is prohibitive. However, whether made synthetically or by fermentation, it remains chemically the same substance, though it is highly possible that a synthetic alcohol, while chemically entirely similar to that made by fermentation, may have different physical properties, as is the case with sugar.

SOURCES OF ALCOHOL.

UTILIZATION OF CEREALS IN THE MANUFACTURE OF DENATURED ALCOHOL.

By H. W. WILEY, *Bureau of Chemistry.*

The subject for discussion this morning is the one which is of most interest to the people of this country, since the production of alcohol in the United States up to the present time has, with a few exceptions, been exclusively from Indian corn. Of course you will understand that pure alcohol is purified by the processes of distillation in chambered stills and by filtering through finely ground, hardwood charcoal to remove certain odors. Neither the chemist nor the taster can distinguish the difference between alcohols made from various sources, at least, when they are first made. It is claimed by some that if the alcohol from any of these sources be kept under certain conditions, perhaps in a warm atmosphere, bottled up and left for some time, weeks or months, an odor will be developed which will make it possible to distinguish one form of alcohol from another, even in a purified state. I have here certain alcohols made from different sources. With one exception, these have not been filtered through charcoal, and I doubt if the chemist can distinguish by careful analysis any difference between them. One is made from the refuse of green corn from Indian corn canneries. The cobs, with some grain attached to them, contain much fermentable matter and this alcohol is made from that refuse. Another sample is made from the green cornstalks—that is, from the stalks at the time of the harvesting of the green corn. These contain large quantities of fermentable matter, and if it were possible to use these stalks in the States of Illinois and Iowa, where the corn becomes hard about the 10th or 15th of September—that is, before it is damaged by frost—they would be found to contain a very large amount of sugar and starch which could be converted into alcohol, and this country could make enough alcohol to furnish all that would be needed for power, fuel, and light. We have about 100,000,000 acres in corn, and these cornstalks would probably average 8 to 10 tons per acre, which would be 200 pounds of fermentable matter per acre—that is, the 100,000,000 acres would furnish 100 pounds of alcohol per acre, this amount of fermentable material going to waste every year. Now, as I have stated, there is absolutely no difference between the properties, taste, etc., of pure alcohol made from green corn and that made

from any other product. A great deal of the waste corn of the cannery could be converted into alcohol. Another alcohol is made from the green corn itself, which should perhaps come first. Thus we have four samples of alcohol made from different parts of the corn crop: The ripened grain (usually a commercial article), the cobs of the green Indian corn cannery, the green Indian corn itself, and the stalks. Thus you see that this one cereal may furnish alcohol, and in commercial quantities, from four different parts or portions of the plant. If Indian corn could be maintained at the price it brought twenty-five years ago, we would not need to consider any other source than the ripened grain itself, for I do not know of any source of alcohol cheaper than Indian corn at 20 cents per bushel. A bushel has nearly 40 pounds, in round numbers, of fermentable matter which would therefore cost about one-half cent per pound. The handling of the wastes and other materials discussed would cost at least one-half cent per pound of fermentable matter contained, if not more.

The next samples to be noted are neutral spirits or alcohol made from cantaloupes, from potatoes, and from waste watermelons. I want to mention particularly the sample made from beet molasses in a commercial distillery in California, which I think is the purest alcohol we have found. I have one more sample of commercial alcohol, a very pure alcohol, made from sawdust by converting the cellulose to dextrose by the action of sulphurous acid previous to fermentation and distillation. As you know, when wood is distilled the alcohol formed is chiefly methyl alcohol, sometimes called "wood alcohol." But when wood fiber of any kind is converted into dextrose by heating with acid, ethyl alcohol instead of methyl is obtained.

We shall now consider more in detail the use of cereals as a source of commercial alcohol. Practically all cereals may be used and have been used in the production of alcohol—rice, barley, rye, Indian corn, oats, and others. The only question which arises in the selection of these materials for industrial alcohol making is economy, as the character of the raw material is of no importance. The waste materials used are the best because the product needs less denaturing. Hence in making denatured alcohol the process of filtering through charcoal is absolutely unnecessary, because any odor which might be present, if left, would partly denature the alcohol. For instance, alcohol made from decayed Indian corn has a disagreeable odor, whereas if made from good corn you could not distinguish any other odor than that of the alcohol itself. For the present purpose the deodorizing process is entirely unnecessary.

It is of interest to know something of the extent to which cereals are used for the manufacture of alcohol for all purposes, and I shall quote from the Report of the Commissioner of Internal Revenue for the fiscal year ending June 30, 1907. Every distillery in this country must keep a daily record of the kind and quantity of materials used and the kind of spirits made. On page 79 of the report a summary of the different kinds of cereals used is given. Unless otherwise specified, the term "malt" in this country seems to imply barley. The quantity of malt used for the year ended June 30, 1907, in the United States is given as 4,440,315 bushels. Wheat is not very extensively used for making distilled spirits in this country, but a notable quantity is employed, the number of bushels being 21,452. Barley, aside from its use as malt, is almost unknown, only 685 bushels of unmalted barley having been used. Rye is used chiefly in the manufacture of whisky, and the amount consumed for the year was 6,250,898 bushels. Indian corn is the principal cereal used for making alcohol, the quantity used during the year being 23,474,509 bushels; oats, 17,301 bushels. The most remarkable fact shown by this table is the insignificant quantity of Indian corn used for distilling as compared with the whole crop. We often think of the distillery as one of the principal markets for our Indian corn, whereas, in fact, it is not the distillery nor even the glucose factory. About 3,000,000,000 bushels of corn were produced in the year 1906, and of that total amount only 23,000,000 bushels were used for distilling purposes. The second most important material used is molasses, the total number of gallons distilled during the year ended June 30, 1907, being 25,722,926; that is, more

than a gallon of molasses for every bushel of corn used for distilling purposes.   Other cereals than those mentioned, such as rice, sorghum, etc., amounted to 4,442 bushels. The average yield of proof spirits per bushel of grain was 4.51 gallons—about 2¼ gallons of industrial alcohol.   The average yield for molasses was 0.52 of a gallon of proof spirits per gallon of molasses.

It may be interesting to know in what parts of the country the distilling industry is most active.   For instance, 10,199,382 bushels of grain were used in Illinois, which is more than that used in any other State, Indian corn, of course, being the largest item. Kentucky is the next State, having used 8,419,770 bushels of grain for distilling purposes.   Indiana follows with 5,649,267 bushels, almost all of which was for the production of alcohol, there being very few whisky distilleries in the State.   Other important States are Ohio, using 2,874,384 bushels of grain; Pennsylvania, 2,446,732 bushels; and Maryland, 1,426,018 bushels.   No other State used over a million bushels, and some none at all.   Louisiana does not distil any grain, but a great part of the molasses made in that State is used for this purpose.   Michigan, Mississippi, and New Hampshire are the only other States having no grain distilleries at all.

All kinds of Indian corn are consumed in the distilleries of the country.   For the making of whisky the finest white corn is preferred.   The Kentucky distilleries are extremely careful in their selection of the raw material.   Indian corn that grows along the Ohio and the Kentucky rivers is especially sought after by the distillers of Kentucky as being peculiarly suitable.   But corn which has been injured by frost, heat, moisture, or mold can readily be used in the manufacture of industrial alcohol as such damage does not much affect the fermentable content of the grain, and it can be bought at a low price.

The composition of Indian corn is very well known, so that one can estimate how much alcohol can be made from a good grade of corn without an analysis.   We have done a large amount of work in the Bureau of Chemistry on the composition of cereals, and in Part IX of Bulletin 13 a number of interesting tables are given, showing the average composition of maize in this country and in foreign countries.   In southern Europe maize is grown very extensively, but not used, so far as I know, for human food.   It lacks the flavor and character of the maize grown in this country and is regarded by the people there as quite unfit for human consumption.   In 1898 I read a paper before the International Congress of Applied Chemistry in Vienna on maize as a human food, and one of the distinguished members of the congress asked me if it were really true that maize was eaten by human beings in the United States.   In 1898 the Bureau of Chemistry issued a bulletin, No. 50, on the composition of maize, which was prepared especially for presentation at the Third International Congress of Applied Chemistry to correct European ideas as to the character and uses of this product.

A typical American maize should have the following composition: Weight of 100 kernels, 38 grams; moisture, 10.75 per cent; proteids, 10 per cent; ether extract, 4.25 per cent; crude fiber, 1.75 per cent; ash, 1.50 per cent; and carbohydrates, other than crude fiber, 71.75 per cent.   The average analyses of the samples of maize collected at the World's Columbian Exposition at Chicago varied very little from these figures.

As has been mentioned, the production of a potato rich in fermentable matter is highly desirable in view of the possible development of this industry in the United States.   But although the production of a maize richer in fermentable matter is quite possible, and investigations have been made along these lines, it appears that we must look forward not to the use of the best quality of Indian corn for alcohol making, but to the utilization of wastes and of other less valuable cereals.   For example, in the Northwest wheat is often injured by early frost, but its fermentable content not much affected, and it could at a low purchase price be profitably converted into industrial alcohol; and so with the other waste or damaged cereal products.

UTILIZATION OF POTATOES IN THE MANUFACTURE OF DENATURED ALCOHOL.

By H. W. WILEY, *Bureau of Chemistry*.

This country is not a potato-producing country as compared with many others. The yield, moreover, is extremely irregular, and for this reason, at the present time, the potato is not to be considered as an economical source of alcohol. When it is realized that a bushel of potatoes is selling to-day in the market for more than a bushel of corn, and that a bushel of corn has 70 per cent, in round numbers, of fermentable substance, and the potato has only 20 per cent, it is evident how economically impossible it would be to use the potato as a source of alcohol. All of the root crops may be considered together, the sugar beet in all its forms, the cassava root, the white or Irish potato, and all the varieties of the sweet potato and the yam. All of these crops have about the same average amount of fermentable matter, the cassava having probably the largest quantity, and the ordinary white potato the least. The sweet potato, the yam, and the cassava root have notable quantities of cane sugar, while the white potato has but little. In regard to the yield of the potato, the crop report for 1907 gives the total number of acres planted as 3,124,000. Compare that with a little less than 100,000,000 planted in Indian corn, and you can see the great difference in these two crops, the average in Indian corn being practically thirty times as great as that planted with potatoes. The total yield of Irish potatoes in 1907 was 297,942,000 bushels, as compared with 2,592,320,000 bushels of Indian corn. The average price per bushel for the whole country in 1907 for the Irish potato was 61.7 cents and that of Indian corn was 51.6 cents, showing that the price of the potato even in a good year is still higher than the price of Indian corn. The State raising the largest quantity of potatoes was New York, with a yield of 41,748,000 bushels. Of the other States, Maine had a yield of a little over 17,000,000; Pennsylvania, 22,900,000; Michigan, 26,900,000; and Wisconsin, 22,700,000 bushels, and others having less, such as Illinois, 13,300,000; Minnesota, 14,600,000; Iowa, 11,900,000; Ohio, 11,900,000, in round numbers. I doubt if any notable quantity of potatoes of any kind was used in this country in 1907 in making alcohol, practically all the alcohol made in the United States at the present time being made from Indian corn.

An interesting report has been issued in the Daily Consular and Trade Reports for Saturday, October 3, 1908, relating to the production of alcohol in Germany, which is the country above all others that utilizes the potato for the production of alcohol. In France the alcohol of commerce is made almost exclusively from the sugar beet, either directly from the beet or from the molasses which forms a by-product of the beet-sugar factories. The cereals in France also play an important part in the manufacture of commercial alcohol. In England commercial alcohol is made largely from Indian corn imported from this country. In Germany last year (1907), according to the report of our consul-general at Berlin, there were 100,218,204 bushels of potatoes used for the production of alcohol—in other words, 2,727,493 metric tons—from which there were produced about 78.5 million gallons of alcohol, constituting 77.3 per cent of the total alcohol produced in Germany during the year, which amounted to 101.5 million gallons. The acreage devoted to potato culture in Germany in 1907 was 8,148,224. In a small country like Germany such an area represents proportionately three times that planted in this country. There were produced 45,538,000 metric tons of potatoes, being an average yield of 205.5 bushels to the acre, while the yield per acre in the United States is only 95.4 bushels, less than half of the yield in Germany. Of the 1907 crop in Germany, 42,618,982 tons of potatoes were sound, while about 3,000,000 tons, or 6.4 per cent of the total, were diseased, or what are called culls in

this country. The American consul-general reports that in 1906–7 there were in Germany 89,601 distilleries. Compared with the number of distilleries in this country, this is a large number and shows the difference in methods followed. Here alcohol is made solely in large establishments, while in Germany it is manufactured largely in small establishments. Of this total, practically 5,640 are under the control of the excise officers, while 83,960 distilleries are not directly controlled, but make their own reports. These may be regarded as agricultural distilleries, but, while Germany uses potatoes chiefly, I want to emphasize particularly the fact that many other materials used in Germany might also be employed in this country—materials which from their very nature could not possibly be used in large establishments because of their lack of value and the cost and difficulty of transporting them any distance. In addition to potatoes, there were about 8,000,000 gallons of alcohol produced from grain, that is, by the agricultural distilleries, and 11,000,000 gallons produced from grain by the industrial distilleries. Molasses was the source of 2,354,736 gallons, which is rather remarkable when you consider that in Germany beet molasses is largely used as a cattle food rather than in the manufacture of alcohol. From wine not suitable for consumption 126,000 gallons of alcohol were made. From the wastes of the brewery 39,784 gallons were produced, and from various other waste materials, 620,379 gallons.

Coming back again to the potato crop, it has been stated that in this country the price of edible potatoes is such that they go directly into consumption, the farmer naturally seeking the largest profits. Therefore only the culls or imperfect potatoes are available for alcohol making. At the present time are used exclusively, as far as I know, in this country for the manufacture of starch. Some years ago the Bureau of Chemistry made an investigation of the starch industry in Maine and published a bulletin on the subject.[a] There were from 15 to 20, or more, starch factories in Maine, especially in northern parts, in Aroostook County, where the potato crop is large, and some factories were solely for the utilization of potatoes not suitable to be sent to market or to be fed to cattle. Michigan is another State where large factories are found, and there are a few in Colorado. The Germans, on the contrary, grow a special potato for alcohol making, and that, in my opinion, is going to be one of the great developments in this country; also the development of the sweet potato and the yam of a coarser structure and larger yield, because the quality and edibility need not be considered, only the quantity of fermentable matter being of interest. Professor Maercker, the famous German chemist, devoted a large part of his long service to agriculture to the development of the high-grade potato, rich in starch, for the purpose of making alcohol. In Farmers' Bulletin No. 268, on the materials from which industrial alcohol may be made, various analyses of German potatoes by König, Lintner, and Wolff are given, showing 20.69, 19.7, and 20.7 per cent of starch and sugar present aside from crude cellulose. The analysis of good Maine potatoes (not culls) by the Bureau of Chemistry showed an average of 18.29 per cent of starch and sugar. In Vermont the starch fell to 14.51 per cent as an average; in New York at the Cornell Station, 19.28 per cent, almost up to the German standard. In France the potato used for industrial purposes averaged 19.006 per cent. This shows that a potato has been developed in Germany which is richer by an average of about 2 per cent in fermentable matter than those raised here, a matter of importance, since it means a 10 per cent increase in alcohol.

Now the development of this industry is going to depend largely on the cheapness of the raw material. The price of denatured alcohol at the present time is necessarily higher than that of gasoline; otherwise there could be no commercial competition on the mere merits of the two articles as produced for heating and the driving of engines. Of course alcohol has great advantages for domestic use, aside from the fact that

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[a] U. S. Dept. Agr., Bureau of Chemistry, Bul. 58.

careful housekeepers would prefer it on account of its greater safety and less offensive odor.   One of the great works of the agricultural colleges and experiment stations so well represented here will be, in my opinion, in the direction of supplying a more abundant and a cheaper source of fermentable matter for the production of industrial alcohol on an economic basis.   Any one of you who has followed the history of Indian corn knows that we shall never have again in this country as cheap Indian corn as was grown a few years ago.   Though the area devoted to the culture of Indian corn has increased and will certainly continue to increase in the future, the price will keep high because of the value of the crop for other purposes.   On the other hand, I believe that the price of the potato, the sweet potato, and the yam will become lower because their production will be so abundant.   For instance, we will be able to obtain an average yield of 200 bushels per acre instead of 95, which will be one step toward the solution of this problem.   We can get 30 or 40 bushels of sweet potatoes more per acre than are produced in England.   Then we have, in the South especially, an area that is called sand lands, which is the tide-water region of the whole coast of the Atlantic to the Gulf coast, running back a considerable distance to rich and hard soil.   These sand lands were originally covered with pine and with very little care and attention will give splendid crops of sweet potatoes and yams. It is evident that the irrigated areas of the West can also yield potatoes in great abundance and at a small cost, even at the high price of the land.   So that, while the potato as a raw material can not now be regarded as of any economic importance for the production of alcohol, it may in the future become, as it has in Germany, of prime importance.

And in the meantime it seems important that we should learn the technics of making denatured alcohol and the treatment of raw materials therefor.   It is also important that at the several stations this problem of extending the source of supply be carefully considered.   Cereals are too valuable for other purposes, but we hope to see developed in this country an agricultural industry in the manufacture of denatured alcohol, not so extensive, probably, as that in Germany, but still of considerable importance. There seems to be no reason why agricultural stills should not be used as well as thrashing machines.   In southern France the still on wheels goes around from farm to farm to work up waste, such as pomace, skins, and grapes not suitable for making wine, beet molasses, etc., and the sugar beets.   And while that probably is not possible nor desirable here because it would be expensive, especially as to fuel, at least we can have one still in each community where available materials are produced in excess on the farms or in the factories.

It seems to me that in the future an important function of the agricultural colleges will be, first, to teach this process of the manufacture of industrial alcohol in the various States, and, second, to study the development of the potato, the sweet potato, and other suitable materials for utilization in this way, as well as the employment of wastes looking to a more economic production.

CASSAVA AND SWEET POTATOES AS SOURCES OF INDUSTRIAL ALCOHOL.

By C. C. Moore, *Bureau of Chemistry.*

In the United States the annual consumption of kerosene approaches 1,000,000,000 gallons.   It is probable that a great portion of this is used in rural districts and that at least 750,000,000 gallons are consumed on farms for the purpose of household illumination.

It is generally stated that 1 gallon of 95 per cent alcohol will equal in candle power 2 gallons of kerosene.   If this is true, then the farmers of this country could use 375,000,000 gallons of alcohol, should they decide to substitute alcohol for kerosene. To make this amount of alcohol would require the following quantities of material:

Of corn, 140,000,000 bushels, necessitating the growing of 5,000,000 acres of this crop, which would be an increase of 5 per cent of that now grown in the United States.

Of potatoes, 450,000,000 bushels, necessitating the growing of 5,000,000 acres of this crop, which would be an increase of 60 per cent of that now grown.

Of sweet potatoes, 375,000,000 bushels, necessitating the growing of about 4,000,000 acres of this crop, or about twelve times as much as is now grown for food consumption and all other purposes.

The requirements of the arts which are at present using alcohol approximate 20,000,000 gallons annually, or about one-twentieth of that which the farmer would use for illumination.

When the farmer uses kerosene he is using a commodity in the production of which he can be in no way interested. When he uses alcohol he is consuming a substance four-fifths of which represents the direct fruit of his labor, the other fifth going for the cost of manufacture. Even with alcohol at double the price of kerosene, the advantage is still in its favor, as the quantity handled would be but half that of kerosene, and this factor is of much importance on the farm where the question of transportation enters into everything.

Should the demand for alcohol be thus increased, the question will not be confined to the utilization of waste products but will rather consist in the selection of the most economical crop that can be grown for this purpose in the various sections of the country. The western supply will probably come from corn; in the North and East, the white potato may enter largely into supplying the demand, while in the South it may be supplied from several sources—molasses, the sweet potato, and the subtropical plant, cassava, which will grow in the Gulf States. Owing to the lack of general information regarding this latter plant, and the possibility that the South, on account of its seaboard advantages, may become a prominent factor in alcohol production, it has been deemed worth while to devote some attention to the possibilities of cassava.

This plant was brought from the Tropics, but it will grow anywhere in the South, and has been grown in the District of Columbia; the length of the growing season, however, is the factor that determines the yield, there being no especial time when the plant should be harvested. A small crop would be harvested after six months' growth, whereas a much greater harvest would be obtained after eighteen months, it being nothing more than a tuberous root which increases in size according to the length of time it is allowed to grow. The crop should not be planted until the soil has become warm, and the growth will cease as soon as it becomes chilled, therefore a growing season would not be obtained farther north than Savannah, Ga., and even at that point would be doubtful.

In appearance the plant resembles a small bushy shrub, about 5 feet high, though the root may be 3 or 4 inches in diameter and 2 or 3 feet long, and frequently there is a cluster of roots of this size. They are very brittle, and as the method of harvesting is to pull the plant it is evident that the soil must be very loose, else there will be great loss through harvesting. The fresh root contains from 25 to 30 per cent of starch and from 5 to 10 per cent sugar, all of which is available for alcohol. It is difficult to arrive at correct estimates as to yields in this country, as the cultivation of the crop has been confined either to a very few garden patches in a soil unusually fertile, or it has been planted and left to grow without fertilization or cultivation. Reports as to the yield have ranged from 2 to 40 tons per acre, according to the enthusiasm of the grower.

The agricultural reports of the British West Indies give the average yield as about 15 tons per acre, the crop being allowed ten months' growth. In Florida, where the growing period is about the same, it is probable that with good fertilization and cultivation a yield of 10 tons would be obtained. This approximates 2.5 tons of starch and one-half ton of sugar, totaling 3 tons of fermentable matter, or a possible

yield of 500 gallons of 95 per cent alcohol per acre. In the South the average price of kerosene delivered to the retailer is 12.5 cents per gallon in bulk, which would indicate that alcohol could sell for 25 cents per gallon wholesale, and compete with kerosene, owing to the general advantages of alcohol for household illumination. Or if computed from the assumed 10-ton yield of cassava, there is $100 per acre, after deducting the cost of manufacture, if based on 5 cents per gallon. This represents the gross proceeds when the raw material is delivered at the place of manufacture.

These data certainly present an attractive proposition, and for this reason it is well to point out the many obstacles that may arise in practice. Among these is transportation, and when shipment is to be to a considerable distance great loss is occasioned through rotting, for decomposition begins within forty-eight hours after harvesting. Rapid work would be necessary, and it is doubtful if a commercial success would result under such conditions. For this reason it was suggested, and experiments were made, to determine the practicability of reducing this and similar products to a more concentrated form, before shipment and for storage, so that the farmer could ship at his convenience and the distiller use the material to greater advantage. The suggestion was to grind and dry the tubers in the immediate vicinity of their production, perhaps using sulphite to arrest fermentation. The apparatus would be simple, consisting of a form of grinding machine made by covering a wooden drum with punctured roofing tin, such that the complete grinder would cost less than $10. This would be run by a gas engine or any suitable power at hand. The grindings would pass on to a canvas-bottom tray, and be run out into the sun, where under good weather conditions and an occasional stirring by use of a hoe the drying would be complete in six or eight hours, after which the material could be sacked and stored. As the crop does not have to be harvested at any particular time, it could be done only as fast as the grinding and drying facilities admitted; and since the drying does not require any particular attention and the grinding would require no more than a couple of hours per day, it is probable the farmer could prepare this crude meal at a cost of less than $1 per ton of meal, representing two tons of fresh tuber. He would thus save 50 per cent of the cost of transportation, could deliver the meal at his convenience, and in addition would have performed one step for the distiller, for which he should be paid.

It might be possible to combine the manufacture of starch with that of alcohol, the latter being the by-product when the material used contains starch and sugar. The United States manufactures about 100,000 tons of starch annually, and from the approximate data here given it is seen that for every ton of starch manufactured from such a material as cassava, there would be waste products equivalent to 100 gallons of alcohol; or, in other words, the starch industry of this country could contribute 20,000,000 gallons of alcohol from its waste products, should the starch supply come from the cassava or a similar plant.

Since this matter is altogether conjectural, and involves the introduction of a foreign plant, it may be of interest to apply the proposed scheme to some crop that is better known, and which can be grown in an area not so limited as is that for cassava. The sweet potato grows in a number of the States, though it is most prolific in the sandy soils of the South. This crop has always had an inflated value, owing to the price that is obtained for it as a winter vegetable, selling in the northern markets for as much as $1.25 per bushel. This is due, however, to the commercial conditions, and not to the cost of production. If a demand should arise for all the sweet potatoes that could be grown as a general farm crop, not as a garden crop, they could be profitably produced for 25 cents per bushel in the States of South Carolina, Georgia, and Florida. The same method as that for desiccating the fresh cassava tuber is proposed for the sweet potato. It is especially applicable to that crop, owing to the peculiar changes that take place during storage, resulting in about one-fourth of the starch changing into sugar. It would not be profitable to manufacture starch from sweet potatoes even at

25 cents per bushel, unless this sugar could be utilized. The best varieties grown in South Carolina contain about 20 per cent of starch and 6 per cent of sugar when fresh. During storage changes take place which result in about 15 per cent of starch and 11 per cent of sugar being present. In the manufacture of starch 12 per cent would be obtained, with a total waste of 14 per cent of fermentable matter. In this instance 1 bushel of potatoes (55 pounds per bushel) would yield 6.6 pounds of starch, which, if valued at 3 cents a pound, would be 20 cents, and the waste product would yield a little more than one-half gallon of alcohol. If the change of starch into sugar was prevented, as it would be if the fresh tuber could be ground and desiccated, there would be a yield of approximately 25 cents' worth of starch and a half gallon of alcohol per bushel. As this is the gross profit, it is hardly probable that the sweet potato would serve as a material for the economical production of alcohol, either independently or in conjunction with the manufacture of starch. As contrasted with cassava it makes a very poor showing, but the comparison is hardly fair, inasmuch as the cassava has been computed on the yield per acre and the sweet potato on the cost per bushel. If the two products are reduced to the same basis—that is, the same quantity of fresh material—the cassava is seen to contain 50 per cent more fermentable matter than the sweet potato, or 1 ton of cassava is the equivalent of 1.5 tons of sweet potatoes. Five tons of sweet potatoes—a very large yield per acre—would be the equivalent of 3.25 tons of cassava. Now the principal items of expense in the production of these two crops are handling and transportation, and given the same amount of fermentable material in two substances, one weighing 5 tons and the other weighing 3.25 tons, it is probable the smaller weight would be one-fourth less expensive to use. This would mean that fresh cassava is worth twice as much as sweet potatoes, for alcohol and starch manufacture, ton for ton. If expressed in yields per acre, 200 bushels of sweet potatoes would be the commercial equivalent of 2.5 tons of cassava, which is a very high yield for sweet potatoes and a very low yield for cassava. The cost of production is approximately the same for the two crops, and any combination of soil, fertilizer, and climatic conditions that would produce 200 bushels of sweet potatoes would undoubtedly produce 10 tons of cassava.

In the manufacture of alcohol from cassava there are two methods of procedure—the ordinary mashing process, and the hydrolysis of the starch by use of acid. The time and apparatus required are practically the same in each case, but in the mashing process the addition of malt is necessary, and this is a factor of decided importance in the production of cheap alcohol. To illustrate, suppose the amount of raw material necessary to make 1 gallon of alcohol could be obtained for 10 cents. For every hundred gallons of alcohol there would have been required from 6 to 10 bushels of malt, if the mashing process is used. Malt has been quoted at $1 and $1.50 per bushel, and 1 bushel of malt produces approximately 2 gallons of alcohol. If the calculation is based on 8 bushels of malt at $1.25 per bushel, for every 100 gallons obtained from the cheaper material, then $10 worth of the cheap material yields 100 gallons of alcohol, and the necessary $12 worth of malt yields 16 gallons, or a total of 116 gallons at a cost of $22, making the raw material cost 19 cents per gallon. If acid is used, there will be required 1 pound of acid for every 15 pounds of starch, or approximately 1 pound of acid per gallon of alcohol. The acid will cost less than 1.5 cents per pound, making the 100 gallons of alcohol cost $11.50, or 11 cents per gallon, as compared with 19 cents when malt is used.

If the illustration be correct, it is evident that in the production of cheap alcohol the acid conversion method should be used, since the use of malt will add 8 cents per gallon to the cost. And in the South, where barley is not grown, the malt would be an expensive item, while sulphuric acid can be had at the lowest rates, and but one-third as much by weight would be required. In addition to this advantage, a large portion of the fiber would be converted by the acid, thus becoming fermentable.

The results of inquiries regarding manufacturing processes may be summarized as follows:

A material such as cassava is cleaned by the ordinary processes, then ground into a fine pulp by the use of some suitable apparatus such as the ordinary millstone, water being supplied if necessary to obtain a pulp of the desired consistency. This pulp could be conveyed directly to a mixing tank containing boiling dilute hydrochloric acid, which mixture should be in the proportion of 1 pound at 18° B. for 30 pounds of fermentable matter, dry basis. The mixture in the tank is then boiled by means of a suitable steam coil, and when the boiling mixture reaches a density of about 21° B. it is conveyed into a suitable copper converter which may be inclosed so as to heat under a pressure of 40 pounds, in which instance the starch and convertible fiber will have been changed to sugar in about fifteen minutes. In lieu of the digestion under pressure, an open-air digestion of perhaps one hour would produce the same change. This converted product is then blown by means of steam pressure into a tank where the acid is neutralized with any suitable material, such as soda ash. The mash is then cooled to about 68° F. and run into fermenting tubs; it should have at this time a density of from 16° to 18° B. Fermentation is then produced by means of yeast, which produces a violent ebullition and a rise of temperature, after which any unconverted fiber is separated by filtration.

If the cassava has been desiccated as suggested and is intended for the manufacture of alcohol only, there should be no occasion for further grinding or treatment, and the material can be unloaded into the acid tanks for conversion or into the mashing tun if it is to be cooked. If the separation of starch is to be made, it will be necessary to soak the meal and put it through a burrstone mill or other suitable grinder. In this case the decanted liquor from the settling vats, or the filtrate from the centrifugals, if these be used, is sent back to be mixed with the original dry meal, so that the first washings, in the continuous process, may be sent to the fermentation tanks, and the second washings of the starch used for the softening and the first washing of the dry meal. Such a process requires the addition of only so much water as is necessary to replace that which has been sent to the fermenting vats. It would be necessary to guard against the bacterial action that may arise, owing to the many forms of enzyms that exist in the plant, and to prevent this the acid which is to be used in the conversion tank could be added at the various stages in the process where the growth of undesirable organisms may occur.

If a manufacturing plant is located in the vicinity of the tuber production, and it is desired to save the expense of desiccation, it will be necessary to store the material that can not be immediately worked, and in which the processes of decomposition may be arrested. In such cases it would be practicable to grind the tubers, as though ready for use and then run the ground mass into tanks, where it could be treated with any enzym-arresting chemical. This would not be an extra process and the macerated product would possibly keep several days.

Three companies have tried unsuccessfully to grow cassava in Florida, but at that time the tubers were used only for starch making and there was a waste equivalent to one-third of the dry matter purchased, which could now be made into alcohol. In addition to this there was great dissatisfaction among the growers owing to the loss of tubers through rotting during transportation. They were shipped to the factory, where each car had to wait its turn in the unloading, which process was coincident with that of manufacture. That is, the car was used for a storage house at the sellers' expense, as the farmer was paid only for the sound tubers finally weighed into the factory. Such a loss would be overcome were the tubers desiccated, and this, in conjunction with the utilization of the large waste product, suggests the desirability of repeating the experiment.

THE POSSIBILITIES OF BANANAS FOR MAKING INDUSTRIAL ALCOHOL.

By G. N. COLLINS, *Bureau of Plant Industry.*

The amount of material available from this source, its distribution, and the cost of production will be discussed in the hope that these data may be of use to those who have the problem of commercial manufacture of alcohol definitely in hand. There are, in reality, two very distinct possibilities: (1) The growing of bananas for the purpose of making alcohol, and the utilization of waste bananas in the field. (2) The utilization of the waste material at the ports of entry.

The growing of bananas on a commercial scale will necessarily be confined to our insular territory, for though they can be raised in southern Florida and in some parts of the Southwest, it is very doubtful if this can be done on a large scale. The Hawaiian Islands and Porto Rico offer the best opportunities. Being unfamiliar with the conditions in Hawaii, my remarks will be confined to the latter locality. The area in Porto Rico that is suitable for banana growing is less extensive than in some of the other West India Islands, but is quite sufficient to establish a profitable industry for the production of this fruit if transportation facilities could be assured. If banana growing be undertaken in Porto Rico with a view to utilizing the fruit as a source of alcohol, it might serve as a demonstration of the possibilities of that island for the production of fruit and force an improvement in the transportation facilities. The manufacture of alcohol and the production of bananas for edible purposes would thus be of mutual assistance.

No information regarding the yield of bananas in Porto Rico is available, but in other regions where similar conditions exist the average yield is from 250 to 300 bunches per acre per annum. Green bunches of good size weigh about 60 pounds each, and with labor at 50 cents gold per day, should be produced at from 10 to 20 cents per bunch. To gain an idea of the value of this material, the composition of the banana may be compared with that of the potato. A complete analysis of a number of varieties of bananas is given in Bulletin 87 of the Bureau of Chemistry, by Messrs. Chace, Tolman, and Munson. This table shows that the per cent of total sugars in the edible portion ranges from 15.36 to 25.66, with an average of 20 per cent. The per cent of edible portion ranges from 62 to 84 per cent, averaging 70 per cent, thus making an average of 15 per cent of sugar in the entire fruit. There is, in addition to this, some other fermentable material, so that the total amount of carbohydrates is almost exactly the same as in the potato. One author gives the following comparative analysis:

Comparative analyses of bananas and potatoes.

Determinations.	Banana.	Potato.
	Per cent.	*Per cent.*
Water	75.71	75.77
Albuminoids	1.71	1.79
Total carbonaceous matter (nonnitrogenous)	20.13	20.72
Woody fiber	1.74	.75
Ash	.71	.97

Since a bunch of bananas weighs about the same as a bushel of potatoes, a direct comparison between bunches and bushels may not be unfair, although the waste would be slightly greater in the case of the bananas.

Unlike nearly all temperate crops, the banana has no definite season, but is harvested throughout the entire year, thus obviating the necessity of storing the material, and permitting the plant to be operated at its greatest efficiency.

There will doubtless be considerable additional expense in establishing and maintaining a plant in the Tropics at a distance from any large base of supplies, but if it should prove profitable to manufacture alcohol from bananas produced at a cost of 20 cents per bunch, the inducement of still larger profits from the sale of bunches of over seven hands for fruit would not be unwarranted.

The second possibility—that of the utilization of waste bananas at the ports of entry—is more restricted, but would appear to be a simpler and more practicable undertaking than the growing of the fruit for this especial purpose. The United States imported during the year ending July 1, 1907, $11,883,168 worth of bananas. This represents about 13,000,000 bunches. Of this amount probably at least 2 per cent is waste material thrown away at the ports of entry. This waste, which must amount to about 8,000 tons per annum, is, of course, distributed along the entire Atlantic seaboard. The greatest amount at any one point is at New Orleans, where approximately 3,500,000 bunches were received last year, the waste of which would amount to 2,000 tons.

This material consists of ripe, rotten, and broken bananas, and is worse than valueless, since it has to be hauled away in barges at a considerable expense. Whether this amount of material is sufficient to warrant the installation of an alcohol plant must be left to those familiar with the requirements of the undertaking. The suggestion, however, has been made that this point would be a favorable location for an industrial alcohol plant, supplementing the waste banana supply by the sugar-cane products.

ALCOHOL FROM CACTUS.

By W. W. Skinner, *Bureau of Chemistry*.

To one who visits for the first time that portion of our country generally designated the "great Southwest" the wonderful growth of cactus is among the first objects to attract attention. In western Texas, New Mexico, Arizona, Utah, Colorado, and southern California, thousands of acres are covered more or less densely with one or more of the several species of this typical arid or semiarid vegetation. One naturally asks, Of what value is this growth? Should this question be put to the native rancher, his reply would be—or at least it would have been until a few years ago—that the stuff was worse than useless. However, the rancher's opinion in recent years has been somewhat changed in regard to the value of this plant, owing to the efforts of the Bureau of Plant Industry of this Department in devising methods for utilizing the material for stock feed. The rancher's opinion of the cactus is due both to its limited value when untreated and improperly handled for stock feed and to its easy mode of propagation, together with its protection of thorns, which prevent its eradication by animals. Given these advantages in the fierce struggle for existence incident to the lack of moisture which characterizes that region, it can multiply at the expense of the more nutritious grasses and plants upon which the rancher depends to sustain his flocks and herds. A crop which utilizes a portion of the scanty rainfall and decreases the available pasture is worse than useless—it becomes a serious nuisance. The utilization of such material for the making of alcohol is worthy, therefore, of consideration, since it would utilize a waste or otherwise unprofitable material.

From the chemical point of view the value of any material for this purpose depends primarily on the content of carbohydrates capable of being converted into sugar, which in turn is capable of being converted into alcohol, so that a chemical analysis of the material is necessary to determine its value for this purpose. While at the Arizona experiment station the writer made analyses of several species of cactus native to southern Arizona, one of which (*Opuntia fulgida*) showed a content of carbohydrate material as high as 16 per cent. Hare, of New Mexico, has found *Opuntia fulgida* to

contain as much as 20.4 per cent of nitrogen-free extract. In the accompanying table is given the chemical analysis of several species of cactus, the interesting feature of which for our purpose is of course the content of nitrogen-free extract. There is also included in the table, for purposes of comparison, the nitrogen-free extract in samples of green corn cobs, green corn, and potatoes.

Analyses of various species of cactus compared with those of other alcohol-making materials.

[Percentage composition.]

Material.	Water.	Ash.	Pro-tein.	Fiber.	Nitrogen-free extract.	Ether extract.
Opuntia fulgida	77.79	4.24	1.60	1.66	14.37	0.34
Opuntia fulgida a	72.63	3.88	.95	1.72	20.39	.43
Opuntia fulgida	81.51	2.21	.96	.83	14.21	.28
Opuntia spinorsior	74.54	4.63	1.77	2.58	15.99	.49
Opuntia spinorsior a	77.37	4.32	1.16	1.97	14.88	.30
Opuntia englemanii	77.20	4.18	.89	2.62	14.72	.39
Opuntia englemanii a	72.60	6.94	.84	3.84	15.37	.41
Opuntia englemanii a	85.75	3.27	.45	1.80	8.34	.39
Opuntia lindheimerii a	77.95	5.81	.92	3.33	11.58	.41
Opuntia lindheimerii	87.36	2.82	.60	1.42	7.54	.26
Potatoes					17.00	
Green corn b					20.00	
Green corn cobs b					20.00	

a Analysis by Hare.   b Analysis by Given.

This table shows that some varieties of cactus contain as much carbohydrate material as do green corn or potatoes. However, the cactus which is probably most available for the purpose, because of its abundance, luxuriant growth, and large yield per acre, is *Opuntia lindheimerii*, which contains only 9.5 per cent of nitrogen-free extract, approximately one-half as much as green corn. Assuming a yield of 1 gallon of 95 per cent alcohol from approximately 13.5 pounds of starchy materials expressed in the table as nitrogen-free extract, it is evident from the analysis given that 100 pounds of *Opuntia fulgida*, showing 20 per cent of nitrogen-free extract, will yield theoretically about 1.5 gallons of 95 per cent alcohol, or about one-half as much as 1 bushel of dry corn, while *Opuntia lindheimerii*, with a nitrogen-free extract content of 9.5 per cent, would yield theoretically 0.7 gallon for each 100 pounds of green material. In any method of handling it would naturally follow that where the whole plant is to be used the most profitable time to harvest would be when the fruits are ripe, or very nearly so. Griffiths and Hare,a in "Tuna as Food for Man," report a sugar content in the fruit of some varieties of 11.7 per cent of the edible portion, and in view of the enormous yield of some species the total fermentable matter at that period is very largely increased.

The data for estimating the yield per acre of the several varieties of cactus are extremely meager. Griffiths,b who has been working for several years with cactus, especially the species generally known as prickly pear, says: "Our knowledge of the yield of prickly pear either under natural or cultivated conditions is as yet quite imperfect and the statements made with reference to yields are made with such reservation as is consistent with the meagerness of the evidence. However, it is believed that all computations in whatever manner made are exceedingly conservative and underestimate rather than overestimate in every case." From observations made by him upon prickly pear planted in rows, but not cultivated, he concluded that the yield at the end of the third year was about 8.5 tons per acre, and from my own observation I am fully convinced that it is entirely possible to harvest fully that amount from

a U. S. Dept. Agr., Bureau of Plant Industry, 1907, Bul. 116, p. 16.
b U. S. Dept. Agr., Bureau of Plant Industry, Bul. 124, p. 25.

the open range in those favored sections where the prickly pear and related forms of cactus with a high starch content grow in great abundance.

Assuming a yield, therefore, of 8 tons per acre with an average content of fermentable matter of 8 per cent (which is less than half the highest value for nitrogen-free extract given in the table of analyses), we obtain as a maximum estimated yield 1,280 pounds of carbohydrate material which, theoretically, is equivalent to a yield of 90 gallons of 95 per cent alcohol per acre. This is, of course, assuming that all of the starchy material is capable of being hydrolyzed, which is probably not true, but so far as I am aware no work has been done to determine this point.

The commercial possibility of the production of alcohol from this source is a matter that can be determined only by direct experimentation, the cost of gathering and handling the raw material being of no less importance than the yield of alcohol. It would seem, however, that the production of alcohol for fuel purposes ought to be profitable in the Southwest, the habitat of the material under discussion, if it is possible to manufacture it at a profit in any part of this country, since fuel for the production of power is one of the serious considerations in this section.

For instance, on the main line of the Southern Pacific in Arizona, coal sells for from $12 to $15 per ton, while the crude distillate brings from 22 to 26 cents per gallon and ordinary gasoline 32.5 to 35 cents per gallon. If, therefore, alcohol can be produced on the spot from a raw material the cost of which would be only due to the handling it would seem to be an inviting proposition, and the product should compete successfully with other fuels for motive power, especially where small units only are required. The refuse from the fermentation tubs, because of its high content of fiber, would be available after drying for fuel purposes in the operations of distilling and would thereby reduce the cost of production.

As to the relative value of the cactus for stock food and for the production of alcohol, it is difficult to make any definite statement because of the many factors entering into the problem. In many places where cactus abounds, for one reason or another it may not be profitably used for feeding stock, and in this section where there is much pumping for irrigation and other purposes, and small engines are used for hoisting, as in mining operations, a cheap fuel made from a refuse material to be had in abundance in the immediate vicinity would seem to have possibilities, though the details remain to be elaborated.

PRICKLY PEAR AND ITS FRUITS AS A SOURCE OF ALCOHOL.

By R. F. Hare, *New Mexico Station.*

The Cactaceæ are a very large family, including several genera which differ from each other very much in appearance, but the name "cactus" does not by any means include as many plants as is generally supposed; in fact, in ordinary usage that name is applied to all thorny plants that are native to the Southwest. For example, it is generally supposed that the factory which was erected in El Paso, Tex., for the production of denatured alcohol used cactus as a source of the alcohol, but such is not the case. Lechuguilla (*Agave lechuguilla* Torr), a plant belonging to the Amaryllis family, and sotol (*Dasylirion wheeleri* Wats.), a plant belonging to the lily family, were the two plants from which an unsuccessful attempt was made to produce alcohol. These two plants are not only very different in appearance from the cactus, but they also differ in their chemical composition. Neither the sotol nor the lechuguilla contains any starch, and the quantity of sugar is quite small. At this factory simply malt treatment was used and consequently only the sugars and starches could be utilized. The plant was erected at a cost of over $40,000, before any laboratory investigations were made to determine the value of either of these plants for alcohol pro-

duction. The failure of the enterprise, due to the low yield of alcohol, was inevitable under such conditions.

The Cactus or Cactaceæ constitute a family of plants which includes several genera that differ much in their general appearance. Of the cactus family probably the plato-puntias or prickly pears are the only ones that might be used as a source of alcohol. The other forms are very similar in their composition, but they grow too slowly. In my opinion it is not practicable to erect a factory anywhere in the United States with the idea of utilizing the wild growth of cactus, for while in some places we have almost impenetrable thickets of this material, it would only be a short time before all the supply near the factory would be exhausted, and its heavy weight and low content of fermentable matter would prevent its shipment any distance. The only feasible plan, then, would be to cultivate the plants in the vicinity of the factory.

If a joint of prickly pear is planted and given any care and cultivation it will be ready for use in from three to five years. In some preliminary experiments at the Government cactus farm near San Antonio, Tex., it appears that about 35 tons can be harvested each year from an acre of prickly pear if the plants are first cultivated unmolested for three years. At this rate of production enough prickly pear can be grown in the vicinity of a factory to supply the demand indefinitely. The analysis of about 100 species and varieties of prickly pear made at the New Mexico experiment station shows an average of approximately 10 per cent of nitrogen-free extract, and probably but little over half of this is in the form of sugar and starch. Hence it will be seen that the amount of alcohol obtainable by malt treatment is quite small. It has been shown that the nonsugar carbohydrates present are very readily converted into sugars by acid hydrolysis, but so far no very successful fermentation of the sugars produced in this way has been secured, and it is probable that they are largely composed of nonfermentable sugars.

The word "tuna" is the Spanish name for the fruits of the prickly pear. These fruits vary in size, shape, and color, depending upon the species and conditions under which they are grown. They are from 1 to 3 inches in diameter and are usually pear or fig shaped, but in some species nearly spherical. They weigh from an ounce to half a pound or more, and when ripe vary from a yellowish green to a dark purple color. There are a great many varieties of prickly pear and the quality of the fruits varies widely in different varieties.

At present it seems more feasible to use the fruits of the prickly pear rather than the stems for making alcohol. The best varieties of these fruits will have from 7 to 14 per cent of sugar, which seems to consist largely of dextrose. This sugar is very readily and completely fermentable into alcohol and, since no previous malt treatment is necessary, the cost of alcohol production would be much less than from the starchy material. The plants usually fruit quite heavily and the yield is very large, the crop obtained from a small plat on our experiment station the past year amounting to 14 tons of fruits per acre, containing at no time less than 7 per cent of sugar, equivalent to 980 pounds, or about 140 gallons, of alcohol. If this sold at 30 cents per gallon, the income per acre from the land would amount to $42. This yield was from a plat on the mesa above the irrigation level that had been planted for three or four years and had received neither cultivation nor water, except the natural rainfall of about 8.8 inches annually. The production of 14 tons of these fruits, yielding $42 worth of alcohol per acre on practically worthless arid mesa land without cultivation or irrigation, looks rather promising.

The present method of gathering the fruits by hand, however, is too slow and expensive, and unless some successful method of harvesting by machinery can be devised the manufacture of alcohol from tunas can not be put on a paying basis. The prickly pear would be about 3 feet high after growing three or four years, and since most of the fruits are formed on the ends of the stems, if a machine is devised that will cut off about one-fourth of the plant from the top it would remove most of the fruits with some of

the stems. A machine of this kind could be made that would harvest the fruits very rapidly, and the stems removed with the fruits could probably be fermented along with them and be made to produce some alcohol. We have harvested the fruits from a small area of the prickly pear in this manner, to see what the effect will be on the way in which the plants form their fruits in the future.

DISTILLERY OPERATION AND CONTROL.

DISTILLERY PRACTICE.

By J. A. Wathen, *Louisville, Ky.*

I am requested to speak to-day on the proper location and construction of distilleries, distilling apparatus, etc., and to explain the operation of such apparatus.

The first point to be considered in constructing a distillery is the question of economy in production. It must be located, therefore, with a view to cheaply and quickly receiving its raw materials as well as to cheaply distributing its finished product, which means that railroad facilities must be sufficient to insure reasonable rates for both raw material and finished product. It naturally follows that the closer you are to the supply of your material from which you produce the distillate and the fuel with which to operate the plant, the better. It is equally important to locate a distillery so that an unlimited supply of good cold water is available, as it requires an enormous amount to operate a distillery, and the colder the water is the more rapidly will condensation take place, and with a minimum consumption of water. It is always best, if possible, to use a water that is soft—that is, one which will not scale your boilers and condensing apparatus. This difficulty, however, can be overcome by treatment of the water. As the average distillery employee is not a skilled operator, the machinery should be of the simplest and most practical construction, especially in houses of medium size and in those that do not operate continuously. A house operating at a large capacity continuously can and does generally surround itself with skilled mechanics, and in such cases the distiller may with profit construct his apparatus and plant with all the complicated labor-saving devices possible, knowing that they will be handled properly and profitable results occur therefrom. To illustrate, let us consider the boiler and its operator, the fireman. By the nature of the work of firing the boiler it is impossible to secure a skilled manipulator, for a man who knows how to fire all kinds of fuel and all kinds of boilers to the best advantage will be a man with so much ability that he will not perform the duties of a fireman. This same rule will apply all the way through in operating the different apparatus in a distillery.

Distilleries should always be equipped with sufficient boiler capacity—rather an excess than a deficiency—for it is important that an even, high pressure of steam be always maintained in order to secure the best results. In small distilleries handling less than 1,000 bushels per day there should be at least three-fourths of a horsepower per bushel of grain mashed, while in very large distilleries this can probably be cut down to one-half.

The machinery should all be strong, erring, if at all, on the side of excessive strength. A distillery in which the machinery and shafting are light and the bearings far apart will operate with a continuous tremor that will in a very short time make heavy repairs necessary and very rapidly depreciate the entire property and mar its successful operation. It is also essential that all exhaust steam and waste heats be utilized in some way; for example, the feed water for the boiler should be taken from the condensing apparatus where the heat of the vapors has been used in partially heating it. All bearings should be ring-oiling, as these utilize the oils most completely.

A distillery building should be substantially and sanitarily constructed. It should be as compact as possible without crowding any department, and that compactness should be directed toward bringing all consumers of steam as closely to the fountain heat or boilers as possible, and thus save loss in condensation by carrying steam through pipes for a great distance. These departments should be so arranged as to be easily accessible and, if possible, at all times in view of the man in charge of the plant.

I will now describe briefly the evolution in the process of producing distilled spirits from grain.

Distillation, according to some authorities, seems to have originated with the Arabs, and came down through the middle ages in the hands of alchemists and monks, becoming extensive in its application only in comparatively modern times. America in its early days naturally followed the primitive methods in the production of its beverage distillates. The early settlers along the eastern coast began to make their whisky from the most common cereals, which were rye and barley malt, and produced what was then and is now known as "rye whisky." Their method of distilling, of necessity, consisted in the use of one copper kettle still, in which the beer was boiled over an open fire and the vapor condensed through a coil; when the beer had all been distilled off, the kettle was washed out, the distillate arising from the beer was put in this kettle, fire applied under it, and the vapor condensed in the process known as doubling, which produced the finished product.

The early settlers or pioneers who came across the wilderness road into Kentucky almost immediately began the production of an alcoholic beverage, and as Indian corn was the principal cereal at their command, this was almost exclusively used. At that time Kentucky was divided into few counties and was yet the province of Virginia, and the first distillery was operated in what was then known as "Bourbon County." Its product became known as "Bourbon whisky," which name has been applied since then throughout the entire State of Kentucky to this type of beverage. The same method of distilling was applied here as in the East. The grain was ground by little water mills or horsepower mills and hauled to the distillery; and, as there were no boilers, the water was heated in the copper kettle, drawn out into little hogsheads and the grain mashed in by hand—thus giving rise to the type of whisky known as "hand-made, sour-mash Bourbon." These distilleries had no fermenters, no means of cooling their mashes by coils, and no machinery with which to do the work. Therefore they were compelled to let these mashes stand in little hogsheads a sufficient length of time to cool to a malting temperature, when the malt and rye was spread on top of the tub, together with some cold water, and again the tubs were stirred by hand and the malt thoroughly mixed in for the purpose of converting the starch into sugar. The mash was then allowed to stand for fermentation and some cold water was added in the little tubs to cool them down to the proper fermenting temperatures. When these mashes had fermented, they were generally dipped by hand into the copper doubler and there distilled. Instead of mashing with hot water, heated in these stills, the spent beer or slop was used to scald the material for the next day's mash.

The next improvement in these small distilleries came with the steam boiler and the first steam-heated beer stills. These stills were made by squaring off two large poplar saw logs with a broadax, planing and digging them out with a foot adze in the shape of a trough and pinning one on top of the other, a large heavy slab of wood being fastened on for a top to the uppermost trough. Beer or fermented mash was introduced in both troughs, into the lower of which steam was run through a pipe leading from the boiler. The beer in this trough was boiled by the heat thus supplied, and its vapors were led through other pipes into the beer in the upper trough. In this way the beer in the upper trough was also boiled, and its vapors were condensed and known as "singlings."

This marked the advent of what is known as a chambered still. The wines from this method of distilling were doubled through the copper kettle, which produced the finished product or whisky. Later these stills were built out of heavy, sawed timbers in a circular shape, with heads separating the chambers—the first being two-chambered, while later a third chamber was added. Next the hand process began to give way before the introduction of mechanical power and the mashing was done either in small or large tubs by machinery. The scalding was effected by both boiling slop and water heated by steam from the boiler.

The next advance was to the continuous still, which is composed of several chambers with charge pipes from chamber to chamber, and with perforations in each head, which permit the steam to enter at the bottom and to pass upward through the perforated heads, boiling the beer as it passes over each one. When the vapors reach the top of the still they pass over through the condensing coil and are converted into singlings. In other words, the beer enters the top chamber of this still, goes over the perforated head down through the charge pipe into the next chamber, and continues this process until the bottom is reached, the steam all the time rising from the bottom, ascending through the perforations and passing out through the condenser, thus making a continual flow of beer into the still at the top and out at the bottom as slop, and a continuous stream of steam entering at the bottom, passing through the perforated heads and out at the top, heavily impregnated with alcoholic vapors. These singlings are also doubled through the copper kettle and are then known as whisky.

At this time coils were used for cooling the mash and the mashing process became a continuous one, scalding, cooling, malting and again cooling to fermenting temperature, pitching with yeast and running to a fermenter to complete fermentation prior to distillation. This completes, up to the present day, the method of production of beverage whiskies.

A great many years ago there was invented in France, I believe, a still for the purpose of rectifying to a very fine degree the spirits produced during fermentation. This method employed fractional distillation, dephlegmation, and separation, and has been perfected until a distillate can be produced so highly rectified as to be commercially free from all secondary products congeneric with the production of ethyl alcohol. This still is known as a "column," is mounted on a kettle, and has attached to it a dephlegmating device commonly called a "goose." The separation between successive portions of the distillate takes place at the tail box according to their different qualities. As the refined alcoholic liquid is desired to be devoid of flavor or character, that quality has ceased to be of any importance in its manufacture, and the only thing considered in its production is quantity. The demand for increased quantity has brought about the invention and practical application of the method of mashing or cooking known as the cooker process. This process of producing spirits in which the cooker and the column are employed is as follows:

The cooker is a cylindrical mashing apparatus and to all intents and purposes is similar to a boiler, with a shaft running through it to which stirrers are attached. When preparing to make a mash the machinery is started and this shaft revolves, water is let into the cooker heated to the proper temperature for mashing, the meal is then let in, steam is applied, and the grain is cooked in this closed vessel to a temperature of 310° F., when the blow-out pipe is opened to reduce the pressure. At this point a vacuum pump is attached to the blow-out pipe (which comes out of the top of the cooker), and when the temperature is reduced to the proper point the malt is added to the cooked mash in liquid form. After allowing a sufficient time for the conversion of starch into maltose, the mash is pumped through cooler pipes and reduced to the proper temperature for fermentation, run into the fermenter where

spent beer or slop is added in a certain proportion together with the yeast, and fermentation is allowed to proceed.

It is essential with this cooker that the mashing temperature of the water should not be too high and that the meal be given enough time to soak up all the water possible. At this time steam should be applied with as much rapidity as possible to carry the temperature up to 212° F., when the valve in the blow-out pipe in the top of the cooker is closed and steam applied at such a rate as will maintain the desired pressure above the mash in the cooker (registered by the pressure gauge), as indicated by the temperature shown on the thermometer that is placed in the cooker below the top of the mash line. Otherwise when the thermometer shows that you have a temperature in the mash of 310° F. the pressure gauge would probably indicate a temperature above the mash line of 350° F., which would be injurious to the mash, causing caramelization of the starch. The cooker process of mashing is advantageous in two important particulars—one is that a temperature is obtained that destroys not only bacterial life, but mold life; secondly, at this temperature the starch is liquefied.

In the distillation of this beer or fermented mash the same process is followed as in the production of whisky up to the time of producing what is termed in whisky distilleries "singlings" or in spirit distilleries "high wines." At this point the redistilling takes an entirely different turn. The singlings or high wines are generally leached through charcoal, after having been reduced in proof, if necessary, to 100; after which they are commercially termed "rectified high wines" or "spirits" (this, however, is a misnomer). These rectified high wines or spirits are then usually treated with an alkaline substance (most commonly soda ash and pearl ash) and placed in the kettle for the purpose of rectification. As before stated, above this kettle is a column, which is a fractional distilling apparatus, constructed with from 20 to 50 chambers, each chamber being connected with the one above by a boiling pipe, through which the steam passes.

Over the top of this pipe is a cap to deflect the steam to the bottom of the head and through the liquid contained thereon. Each chamber is also provided with a charge pipe, which extends above the head about 6 inches, so as to guarantee at all times this depth of liquid on each head, through which the steam must pass. Any additional liquid accumulating upon these heads passes downward through the charge pipes from chamber to chamber until it "tails back" from the bottom chamber into the kettle.

Above the top of this column is a dephlegmating device known as a "goose," through which the vapor passes, the pipes of which are surrounded by water at a temperature of about 170° F., which condenses all vapors that boil at a higher temperature. Attached to the bottom of these vapor pipes in the goose are drain pipes, which carry the condensation back into the top chamber of the column, to be again subjected to fractional distillation. All vapor that is not condensed by this goose or dephlegmating apparatus passes over to what is known as the "condenser," which is kept at a very low temperature and condenses all the vapor passing from the goose. This condensed distillate runs out into what is known as a "tail box," which has leading from it several different pipes, that in turn go to different tanks, in what is known as the "wine room." As there are, commingled with ethyl alcohol, aldehydes and other alcohols of a higher boiling point, and as the aldehydes in a free state evaporate at a temperature slightly less than 70° F., it is patent that the first distillate coming over will be most heavily charged with aldehydes. In fact, the first running of the column will be so wholly charged with aldehydes as to keep the proof below the commercial alcohol strength, which is 188 per cent, and until the proof at the tail box reaches this degree of strength, the product is turned into the pipe leading to what is known as the "low wine tank." When the proof at the tail box has reached 188 per cent, the flow is changed into the pipe leading to the alcohol tank.

This flow will continue into the alcohol tank for some time, or until the product is sufficiently free from aldehydes to be of too high grade for commercial alcohol, but which has not yet reached that degree of purity to be termed "neutral spirits." At this point the flow is again changed, and the product run through the pipe into the tank known as "No. 2 spirits," which are mixed with the rectified high wines for the production of spirits in the next change. After it has run sufficiently long as No. 2 spirits to be freed of its aldehydes, the flow is then changed to the neutral spirits pipe flowing to the cistern room. The proof of this delivery is maintained at from 190° to 196°. This is continued until the strength is out of the kettle and at least half of the lower chambers of the column show nothing but water.

About at this point the spirit is in danger of becoming foul by the forcing over of some high-boiling alcohols, and therefore the flow is changed again into the alcohol tank and what is known as "tail alcohol" is run off. When the strength of the distillate becomes too weak at the tail box for alcohol, a small quantity of low wines is produced. By this time practically all of the ethyl alcohol has passed over, and there will have gathered in the top chamber of the column the higher boiling alcohol, commonly called "fusel oil." Most columns are constructed with what is called a "by-pass" at the top, and when the fusel oil begins to come over this is opened, and the fusel oil passes over and is condensed and carried to the fusel-oil tank.

A thorough distiller will redistil his alcohol and recover therefrom a considerable quantity of high-grade spirit. The balance of the product from this distillation will be low wines, and he will redistil these, from which he will recover more low wines and commercial alcohol, which is sold to the trade as such. However, some distillers never recover any spirits from their head and tail alcohol, but sell it all in conjunction with the alcohol produced from the low wines to the trade as commercial alcohol. In order to produce a neutral spirit from the wines of fermented grain, it is necessary to employ fractional distillation, dephlegmation, and separation.

I have omitted to call your attention to one important question in the operation of a distillery, namely, the milling system. The object of scalding grain is to dissolve or rupture the starch cells and produce a perfect starch paste. The more perfectly these cells are ruptured and the starch is pasted, the more perfect will be the conversion of starch into maltose, and the greater will be the yield therefrom, therefore the more finely ground they are in the process of milling, the more perfect will be the results from the distillery. Furthermore, a smaller quantity of malt will be required for hydrolyzing, for when the starch granules are not thoroughly acted upon in the mash tub at the proper temperature for malting, it will be necessary to malt in an excess of diastase to act upon the starch that will be set free during the process of cooling and fermentation. This excess of diastase will not only hydrolyze starch, but will have a tendency to strengthen the growth of the yeast and produce more enzyms, which have both a hydrolyzing effect upon starch that is unconverted, and also inverts or further reduces the malto-dextrins into completely fermentable sugar.

YEASTING.

By J. A. WATHEN, *Louisville, Ky.*

Proper management of the distillery—that is, perfect control of man and apparatus—is necessary in order to get the best possible results. A man may be a very fine chemist, perfectly able to produce a pure-culture yeast of the isolated variety and even of the very finest attenuating species, yet be a failure from the practical standpoint in operating a distillery. It is very essential that the man who directs the operation of the distillery be not only familiar with the technical and scientific detail, but also competent to take hold of the apparatus and do better than the man he is directing. He must be able to operate the machinery, to direct the firing of

the furnace, to operate the stills of all descriptions, the mash tubs, and the cooker; otherwise he will not be able to judge whether they are doing it properly. He must also know this in order to instruct the men under him how to do their work intelligently and correctly, for he will find in practice that the distillery workers are not made up of scientists or specialists in any line. Another object of this is to insure good discipline in the house, for wherever the man who is directing the force of a distillery has more knowledge than his employees he will command accuracy and intelligent manipulation. My experience leads me to believe that the average workers around a distillery are very prone to take advantage of those who do not know more than they do.

It is further necessary in order to properly control a distillery to know from the results just what has been done. This has two objects. When your employee knows that by tests you can check the accuracy of his work he will be careful; again, should a mistake, intentionally or otherwise, be made, it enables you to locate at what point the error occurred, and thereby know how to prevent it in the future. To this end a chart should be prepared and kept with as much care as a trained nurse would keep the chart of a patient in a hospital.

It is important to know the class of material you are handling—that is, the amouns of sugar there is in the raw material. It is hardly possible in practice to make an analysis of the sugar in each kind of grain. Very few distilleries are equipped to do this work, but by testing the moisture of the grain and determining the weight per bushel a good commercial knowledge of the value of the material may be obtained. It is very plain that a grain having 20 per cent of moisture will not produce the amount of sugar that another having 12 per cent of moisture will yield, and one weighing 50 pounds per bushel will not be so good as one weighing 56 pounds. So, for practical purposes, this is a sufficient test.

It is then necessary to know the proportions of water and grain in your mash tub or cooker in order that the degree of conversion may be checked when the mash it tested for saccharine.[a] This content of saccharine, formed during mashing, is affected, as you have all noticed, by the percentage of moisture, by the quality of the material as indicated by the weight per bushel, and by the fineness or perfection with which the meal has been prepared for the mashing process. If one finds after testing that the saccharine content is not what it should be, taking into consideration the quality and moisture of his grain, he must then determine whether the grinding of the grain, the temperatures employed in converting and scalding, the time of standing for conversion, or the quality of the malt is at fault.

It is important to know the saccharine and acid content of the fermenter when pitched in order that the conditions of fermentation may be understood. A fermenter, therefore, should be thoroughly agitated when filled and pitched with yeast, and the saccharine and acid correctly determined. At least once every twenty-four hours this test should be repeated and finally made when the beer has gone to the still. In a house that is running to the best advantage a thorough attenuation and a small increase of acid will be found. A slow attenuation and an undue increase of acid invariably indicate that bacterial ferments are predominating over the true ferment or yeast, and by making these tests at regular intervals this injury may be detected at once. The earlier it happens the worse it is for the result, as after a large portion of the sugar has been converted there is less material left to injure.

It is important to know in double distillations, especially in small distilleries, the yield in high wines per bushel of grain mashed. After this has been ascertained, and knowing the amount of saccharine that has been converted into alcohol, it may be determined whether the operator of the beer still has used due precaution in the proc-

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[a] This term is used commercially to cover all fermentable sugars.

ess and extracted therefrom all of the alcohol, and if he has used due care it will indicate whether there is a leak in the distilling apparatus. When these wines are doubled, it is essential to know the yield of finished goods in order to detect any flaw in the distilling apparatus or its operation. Too great a difference between the high-wine yield and the finished yield indicates imperfection of apparatus or inattention of the operator. At the receiving room where the goods are barreled and weighed by the Government the barrel yield is obtained. If that varies too much from the finished yield, it indicates that there is a loss between the distillery and the receiving room. By this series of checks one may efficiently operate one or an unlimited number of distilleries, because you can bring the condition of any distillery before you on paper, see every move that has been made, and know who has done the work incorrectly, or what apparatus is inefficient. It is equally as necessary to have such a record if you are at the distillery yourself, because no man can be at all points at the same time and see all the work done.

The operation of mashing has been considered in the previous lecture (see page 111), so we will pass to the consideration of yeasting.

There are three common methods of preparing a pitching yeast. The one generally employed is known as the Hungarian method, in which certain proportions of rye and malt are mashed in enough water to give a very thick mixture, permitted to stand a certain length of time for the lactic acid to form, and is then fermented and used in the fermenter as a pitching yeast. There is a yeast commonly used in this section of the country in making the eastern rye whiskies which is made on identically the same plan as the Hungarian except that hop water is employed in the mash, and it is not permitted to stand over to take on lactic acid, but is fermented over night the same as in the process employed in fermenting or working down the sour wine or Hungarian yeast. This yeast acquires a slight amount of acidity, however, during fermentation.

The next procedure is one in which the yeast is mashed by the same method as in the preceding cases, thinned to about the consistency of a rye fermenter with water, stocked with a malt dona, permitted to work two or three hours, and used immediately in the day's mashes. This is commonly known as hop yeast and must always be stocked with a dona. Two methods of producing the donas with which to ferment the pitching yeast are followed. One is to make a mash as in the Hungarian process, using a very thick mixture of rye and malt (which has been permitted to lie over and sour or not, at the option of the yeast maker), cool it to a proper pitching temperature, stock with a fountain yeast, and ferment for from fifteen to twenty hours. This dona is then employed for stocking the yeast which is used to pitch the next day's mash. The other method is to make a pure malt mash of a density of about 20, the water of which has been hopped. Allow it to stand long enough to convert the fermentable matter, cool to a pitching temperature, and stock with a fountain yeast. After working for from fifteen to eighteen hours, it is employed in stocking the yeast, which is used to pitch the next day's mash.

Various methods of producing the fountain yeast are employed. It is very common in Europe and is also becoming a general practice here to employ what is known as a pure yeast culture made from an isolated cell, the method for producing which has been brought to a considerable degree of perfection by Hansen. It is especially adapted to breweries. A brewer strives to obtain a peculiar flavor for his product and to maintain that characteristic evenly and at all times. In making industrial alcohol we are striving to produce the greatest quantity of alcohol from a given quantity of meal, and the essential point is attenuation. There is a doubt in my mind as to whether the isolated-cell theory is at all times advantageous for this purpose. Certainly it should not be employed indiscriminately, and the cell should be tested

thoroughly in comparison with others to ascertain its attenuating power. The old method in this country was to prepare a malt wort, sometimes a mixture made from all kinds of grain, and allow it to ferment exposed to the atmosphere (what is commonly known as spontaneous fermentation); then to make another similar wort and pitch it with this first production of yeast (commonly called spontaneous yeast). There was very little care used in the production of this wort to keep it from being unduly exposed to contaminations. There was also very little knowledge displayed in mashing or preparing the wort so as to favor the growth of the yeast spore, with the result that these yeasts were very heavily contaminated and often entirely overpowered by other germs. Neither the old method nor the isolated-cell method seems entirely satisfactory. I aim to produce a wort that is a perfect medium for alcoholic fermentation; that is, one that is the least liable to invasion by bacteria and the most favorable to the growth of the best yeast spores. The wort is sterilized thoroughly and exposed until fermentation begins, when further invasion by the atmospheric bacteria is prevented by sealing it. Another wort, similar to the first, is then prepared, sterilized, and sown with a very minute amount of the first production, which, under the microscope, will show a considerable number of bacteria, though not an alarming amount, the yeast being practically uniform. I pitch 20 gallons of this specially prepared wort with 6 ounces of the first production, which is a very small amount, in a very large mass. The germs that are in this small quantity of yeast are so overwhelmingly yeast spores that they immediately take hold, and the bacteria are practically dormant for the reason that the wort is not favorable to their better growth, but is to the growth of the yeast spore, with the result that at the end of fermentation (again applying the microscope) you can detect practically no living germs other than this peculiar type of yeast. I am using to-day a continuation of the yeast that was started in 1900 by this method. It has the same appearance under the microscope and has equally great attenuating power as it had at first. There has also been used side by side with it in the same distillery an isolated-cell culture, and a most excellent one judging by actual results, yet it possessed a peculiarity which gave a fermentation of 2 feet of foam, whereas the spontaneous fermentation gave only about an inch. There being no difference in the yield, the advantage was with the cell that I had cultivated, as it is a dangerous thing in a distillery to have a high fermentation.

If the must of wine is exposed to the atmosphere and permitted to gather the germs, you will not obtain the ordinary yeast spore, or *Saccharomyces cerevisiæ*, but you will get the wine yeast spore. Likewise I have prepared a second medium made in a different way from the first and have produced an entirely different spore, the appearance of which is no more like the ordinary yeast spore than is a white man like a negro, nor does it produce the same flavor in the distillate. These two experiences with the wine yeast and this special yeast have led me to believe that by preparing the medium best suited to the growth and nutrition of the spore desired, you will attract and cultivate the strongest and best type of that variety to the disadvantage of all other germs and by perpetuating the germs thus obtained you will soon perfect a pure culture best adapted to the purpose in hand.

This theory is based on the idea of the survival of the fittest, but I may be mistaken in it. It was generally accepted until lately by most practical people that a yeast could not be generated for more than four or five generations. This seemed to be based on the idea that, as the continued reuse of land for the same crop weakens its producing power, so a resowing of yeast would cause a weaker growth of each successive generation. This idea was clearly an error, as my experience has amply proven, for I have carried a regeneration on for more than 1,000 generations with no diminution in the power of the yeast whatever. This may be because in the growth of yeast we do not resow in the same field, but prepare a new one each time.

CHEMICAL CONTROL OF DISTILLERY OPERATIONS APPLYING ESPECIALLY TO GRAIN DISTILLERIES.

By A. M. BRECKLER, *Louisville, Ky.*

The object of exercising chemical control in a distillery is to obtain the largest yield possible by insuring a complete fermentation and preventing a loss of alcohol by acetification or in the still. The first requisite is to test the raw material for the amount of extract contained, which, with the exception of a small and constant percentage, is fermentable carbohydrate.

The methods are as follows:

Heat 300 cc. of water to 65° C., stir in 50 grams of malt, and keep the resultant mixture at that temperature for exactly one hour with constant stirring. Make the mixture up to 450 grams at the end of this time and filter, returning the first 100 cc. to the funnel. The per cent of extract is then found from Table III, page 209, Bulletin 107, Revised, Bureau of Chemistry, by taking the specific gravity of the filtrate at 15° C. The malt must be ground so that it will all pass a 20-mesh sieve. To determine moisture two grams of the malt so ground are dried at 100° C. for two hours. The moisture should be under 7 per cent. The formula for computing extract is as follows:

$$\frac{800 + \text{per cent moisture in malt}}{\dfrac{100}{\text{per cent extract in filtrate}} - 1} = \text{per cent extract in malt.}$$

Raw grain.—Grind 26 grams to 20-mesh and use 25 grams. Bring 300 cc. of water to 40° C., and stir in the grain so that no lumps are visible, bringing the mixture to boiling as soon as possible. The mass is stirred from the start until it has boiled one hour, the water evaporated being replaced from time to time. It is then cooled to 65° C. and 25 grams of malt, whose extract has been determined, are stirred in. The mixture is then kept at 65° C. and stirred for one hour, after which it is filtered as in the case of malt. The extract and moisture are determined as before. The following formula gives the per cent of extract in the grain:

$$\frac{1600 + \text{per cent moisture in grain} + \text{per cent in malt}}{\dfrac{100}{\text{per cent extract in filtrate}} - 1} - \text{per cent extract in malt} =$$

$$\text{per cent extract in grain.}$$

The equations given are for use with a table of reciprocals, and give the results with the use of only one multiplication. The method may not give the total extract, but it does give the total extract available for fermentation, which is what is wanted. The following grades may be taken as outlined by the variations in distiller's extract determined in this manner:

Grades of materials based on determination of extract available for fermentation.

Quality.	Malt.	Corn.	Rye.
	Per cent.	*Per cent.*	*Per cent.*
Extra	68	70	69
Good	66–68	68–70	67–69
Fair	64–66	66–68	65–67
Poor	64	66	65

Besides the extract, the value of malt lies in its power to convert starch into sugar. The following method is used to determine this factor:

(1) *Preparation of soluble starch.*—Genuine potato starch is soaked for ten days in 7.5 per cent hydrochloric acid. It is washed by decantation with distilled water until

the decanted liquid indicates about 0.01 normal acidity. Sodium bicarbonate is added a little at a time until the solution when titrated against standard acid is 0.01 normal alkali to methyl orange, being careful to shake after each addition of bicarbonate. The starch is then washed with distilled water until free from chlorids, when it is tested in the following manner: An amount of mixture containing approximately 10 grams of starch is used. It is boiled, the soluble starch precipitated with chemically pure alcohol, and the clear liquid evaporated to about 2 cc. No precipitate should form on the addition of silver nitrate. Eight to twelve washings after the bicarbonate is added usually suffice when starch and wash water are 1:10. This starch is then filtered off on a Buchner funnel and the resulting cake shaved fine and dried to from 4 to 6 per cent moisture at a temperature under 40° C.

(2) *Determination of diastatic power.*—Twenty-five grams of malt, ground to pass a 20-mesh sieve, are weighed, placed in a flask with 500 cc. of distilled water, and kept at from 25° to 30° C. for six hours, shaking every half hour. The extract is filtered, rejecting the first 100 cc., and 5 cc. of the filtrate are made up to 60 cc. Meanwhile a 2 per cent solution of soluble starch has been prepared and 10 cc. measured into each of six tubes. These are brought to 25° C. and into them is measured, successively, 0.6, 0.7, 0.8, 0.9, 1.0, 1.2 cc. of diluted filtrate. After standing one hour at 25° C., 5 cc. of Fehling's solution are added to each tube and it is placed in boiling water. After remaining twenty minutes they are all removed and the value of the malt determined as follows:

If all the tubes are reduced, the malt has formed over 15 times its weight of sugar; if 5 are reduced, over 12.85 times its weight of sugar; 4 indicates over 11.25; 3, over 10.00; 2, over 9.00; and 1, over 7.50. These values are usually reported as 1,500, 1,285, etc. The following grades are thus determined: Extra, 1,500; good, 1,285; fair, 1,125; poor, 1,000; very poor, 900; worthless, 750 and below. Any malt below 1,285 is the result of poor selection of the original barley, or careless growing.

Having briefly outlined the testing of raw materials, the distillery operations should be considered. The first loss which can occur in a distillery is through faulty grinding in the mill room. This can best be checked by making an extract determination on the grain as ground in the mill and then on the product as reground in the laboratory. It will be found that not more than 1 per cent of extract is lost in this manner when the mills are being properly handled; a 2 per cent loss shows poor grinding which must be obviated by resetting the rolls or sharpening them, matters usually left to the millwright.

From the mills the grain passes to the mash tub. The corn is there doughed in at about 65° C. and cooked rapidly up to 100° C., the object of this cooking being to gelatinize the starch. To do this it is generally necessary to hold it at 100° C. for some time. About twice a week, or oftener, if buying grain in small lots, samples are taken of a mash at ten-minute intervals after it has reached 100° C. These samples are each diluted 1:20 and a few drops stained with iodin on a slide. The starch granules ungelatinized appear as dark blue specks when examined with an inch objective. These should not be present to any great extent after twenty minutes' cooking. If they persist for thirty minutes, a few pounds of the malt should be weighed in with the corn to thin out the mash somewhat and permit of a more intimate contact of starch and steam. The mash, after the corn is cooked, is rapidly cooled down to 64° C. and the malt is added to convert the gelatinized starch. The conversion of starch into sugar now takes place. This conversion is tested after twenty minutes by diluting the mash 1:20 and filtering until the liquid runs through fairly clear. On the addition of a drop of decinormal iodin solution to 20 cc. the color should not appear violet. A few drops more are added, one at a time, with careful stirring, and no evidence of soluble starch should appear. The mash is tested again after thirty minutes if conversion is not complete in twenty. If the conversion is not complete in thirty minutes the temperature of conversion is wrong or not enough malt is being used; usually the latter is the difficulty. The thermometer is compared with a standardized thermometer. The

malt should be present in such an amount that the product of its percentage in the mash and the diastatic power is at least 13,000.

From the mash tub the mash goes to the coolers and thence to the fermenter. The mash while running into the fermenter is sampled and the direct per cent by weight of extract determined by a Balling hydrometer. The mash being very viscous, it is necessary to dilute with an equal volume of water and calculate back to the original sample. Saccharimeters are available which run from 0 to 10 in tenths and have a stem 10 inches long. The readings can easily be made to one-twentieth per cent. Another sample of mash is diluted by weight until 0.5 per cent of extract is present. The maltose is then determined in this solution and when calculated back to the original mash should be 73 per cent of the total extract. The theoretical value is 80 per cent, but owing to the presence of solids not sugar the figure quoted is very satisfactory. This determination affords a further test of the quality of the malt, as malts giving below 70 per cent of maltose are not of good quality, provided the correct temperature has been used in the mash tub. This test need only be made when starting on a new lot of malt.

The specific gravity of the yeast, the mash, and the slop is taken before they go to the fermenter. A 20 cc. portion of each is titrated with decinormal alkali, using phenolphthalein. In the case of yeast 3 cc. are allowed for carbon dioxid in solution. The following are permissible limits:

Permissible limits for acidity and saccharimeter readings.

Determination.	Mash.	Yeast.	Slop.
Acidity [a]	0.01–0.015 normal	0.14–0.15 normal	0.05–0.07 normal
Balling	15–17	7–9	2–3

[a] The acidity is here expressed as normal concentration.

After the fermenter is filled it is agitated with air and a sample taken. The Balling readings should then be from 10 to 12 and the acidity from 0.05 to 0.06 normal. If this is not the case, the desired result is obtained by manipulating the water content of the mash within the limits given, leaving out some slop and filling up the fermenter with water, or increasing or decreasing the amount of yeast or mash according to which is at fault. The importance of a careful starting can not be too much emphasized if constant results are desired.

Having set the fermenter each day at noon the acidity and the per cent Balling are determined. The following table gives what may be considered permissible limits for corn mashes, the exact figures being to a great extent dependent on the outside temperature:

Limits on acidity and Balling determinations for corn mashes.

THREE-DAY BEER.

Determination.	First day.	Second day.	Third day.	Fourth day.	Fifth day.
Acidity [a]	0.04–0.05	Below 0.065	Below 0.075	Below 0.085	
Balling	10–12	2.5–4.0	1–2	Below 0.5	

FOUR-DAY BEER.

Determination.	First day.	Second day.	Third day.	Fourth day.	Fifth day.
Acidity [a]	0.04–0.05	Below 0.063	Below 0.07	Below 0.078	Below 0.085
Balling	10–12	4–6	2–3	1–2	Below 0.5

[a] These concentrations are expressed as normal.

Three-day beer is that which has been set the first half of the week and, under existing United States internal-revenue regulations, must be distilled on the fourth day inclusive from the filling of the fermenter, Sunday not being counted. The beer set the last half of the week is distilled the fifth day, inclusive. It will be noticed that the apparent attenuation is very great at first and becomes less at the end. While this is mainly due to the slackening of fermentation, it should not be forgotten that any such effect is intensified by the lowering of the specific gravity and hence of the apparent Balling, due to the formation of alcohol.

On the last day the beer is tested to show whether the fermentation has been conducted properly and what the yield of spirits ought to be. The Balling and acidity are determined as usual. Twenty cubic centimeters of the beer are made up to 100 cc.; 80 cc. are distilled and the distillate titrated. The volatile acidity should not exceed 15 cc. N/10 acid per 100 cc. of original beer; if it is, the lactic acid mash has become infected in the yeast tub or some infection has been introduced in the mash by unclean pipes and fermenters. The cause should be carefully traced. It might be said that such trouble is usually due to insufficient lactic acid in the yeast and can be predicted from a low acidity of the original yeast mash. The dextrin is determined and should be less than 0.3 per cent of the beer. The alcohol is determined for the purpose of checking the results in the still, but this will be discussed later.

Having considered the mashing and fermentation, we will take up a subject which, while not strictly chemical, yet is necessary for the interpretation of chemical determinations, namely, the temperature of setting the mash. In the summer time, for the greater part of the fermenting period, the outside air warms the contents of the fermenter above the temperature that it would naturally reach by the action of the yeast alone. In the winter, the cold air acts in the opposite manner. By warming, the metabolism of the yeast is increased so that it finishes its life history very quickly; by cooling, its action is slowed so that it takes a comparatively long period to reach the condition of maximum vigor. If the fermentation of the yeast is soon over, the acetic acid bacteria, having nothing to restrain them and being favored by the warmth and humidity of the summer air, gain the upper hand and during the remaining period before distillation cause enormous loss of alcohol. On the other hand, in the cold weather, the yeast being so long in reaching maturity, the attenuation becomes slow and fermentable matter escapes in the slop. It is usual then to set at as low a temperature as possible in summer, 17° to 19° C. being used, and in winter at 20° to 23° C. It is found impossible in most distilleries to set below 17° C. in summer. Now if the volatile acidity and the total acidity are too great and the initial acidity is correct, the most probable explanation is too high a temperature. If the acidity is correct and the beer is still not attenuated the reverse is true, so that the chemist must determine these points and see that the temperature is raised or lowered accordingly. Following is a table of temperatures from averages taken from the laboratory records of the distillery:

Average temperatures of the mash in summer and winter.

SUMMER (OUTSIDE AIR 29° C.).

Age of fermented mash.	First day.	Second day.	Third day.	Fourth day.	Fifth day.
	°C.	°C.	°C.	°C.	°C.
3-day beer	19.5	28.5	31.5	29.5	....
4-day beer	18.5	26.5	30.0	31.0	28.5

WINTER (OUTSIDE AIR 5° C.).

3-day beer	23	29.5	31.5	28.5	....
4-day beer	22	27.5	32.0	31.0	27.5

In the case of the three-day beer the maximum temperature has been passed between the second and third days, while for the four-day beer it generally is passed between the third and fourth days. The maximum temperature reached in both cases should not be over 32.5° C.

Having finished the fermentation the beer is next run to the still. The general opinion of practical men is that here some alcohol is always lost in the spent beer or slop. These opinions are based on the rather uncertain indications of the slop tester, an instrument which condenses a small portion of the vapor from the lower or from the second chamber of the still and passes it into a cylinder in which a delicate hydrometer is placed. It is my experience that the separation is absolute. In order to test this a sample of slop is collected every ten minutes during the distillation. About 100 cc. are drawn off from the bottom chamber of the still through a cock provided for that purpose and run into a tin can with tight-fitting cover. Ten such samples are collected in the same can, thoroughly cooled and stirred; 500 cc. are measured out at from 15° to 20° C. and washed into a liter distilling flask with about 200 cc. of water and 100 cc. are distilled. The distillate is tested for alcohol by the iodoform test or the molybdate test as given in Allen's Commercial Organic Analysis, vol. 1, p. 90. A blue color in the latter case is formed, by the intensity of which the approximate percentage of alcohol present can be estimated. Warning must be given that no slop should be allowed to carry over into the distillate as all carbohydrates give both reactions. If both tests show the presence of considerable alcohol, a quantitative test must be made. As many methods for alcohol determinations are impractical, the following is given as one easy of manipulation and quite rapid. It is used for slop, beer, or any other alcoholic liquid tested in the laboratory.

Seventy-five grams of material are weighed in a 500 cc. flask, a nitrogen trap and stopper placed in it, and connection made with a coiled condenser by a short piece of rubber tubing. To the end of the condenser is connected a glass tube drawn down to a capillary. The capillary reaches to the bottom of the standardized 50 cc. flask. A mark is made on the side of this flask at approximately 40 cc. When this amount has passed over, the capillary is lifted from the flask and washed off outside and inside with about 6 cc. of water, receiving the washings in the flask. The volume is made up with cold distilled water after the flask has stood one-half hour in water at 15° C. The flask is then taken out and left to come to room temperature, when it is weighed against a counterpoise. Results on specific gravity check to 0.00002. The alcohol table of Hehner given in Leech's Food Analysis and Inspection should be used as it corresponds exactly to the table in the United States Internal Revenue Gaugers' Manual for low percentages.

If there is alcohol in the slop, it is of course a dead loss, and the still must be gone over thoroughly by a coppersmith and the man handling it carefully watched. The alcohol found in the beer should check within 0.5 per cent with the alcohol extracted by the still. The effluent from the still or slop should not show over 3 per cent Balling, a higher value denoting incomplete fermentation. The acidity should not be over 0.085 normal.

It might be said in conclusion that these tests are the simple and reliable ones which furnish the routine work in my laboratory, where as many as 40 samples a day are tested for temperature, acidity, and Balling. The complex tests and details belong more properly to a lecture on theoretical distillery practice. The simplicity of those given is their chief recommendation, all 40 samples being tested by myself and assistant, the utensils cleaned and put away, and the results entered in a permanent record in three hours.

PRACTICAL YEASTING OF DISTILLERIES, WITH SPECIAL REFERENCE TO GRAIN DISTILLERIES.

By A. M. Breckler, *Louisville, Ky.*

In this lecture the method of yeasting in a large distillery and some simple bacteriological examinations which can be made by any chemist without special training will be presented. The growth of any kind of bacteria requires a favorable medium. A formula for furnishing such a wort or medium for yeast is briefly as follows:

Boil 2 kg. of sound hops in 150 liters of water for one hour. Strain off the clear liquid through a colander, cool to 82° C., and stir in 50 kg. of barley malt. After standing four hours, run off through a sieve until it comes out clear. Then return the cloudy portions to the original vessel and allow them to come through with the rest of the liquid.

In order to bring about fermentation, the presence of yeast is necessary. The older distillers depended on the spontaneous impregnation of miscellaneous secret mixtures by yeast cells from the air, the peculiar ingredients of such a mixture being supposed to possess certain beneficial effects on the resulting fermentation. The modern practical distiller no longer depends on chance, but insists on impregnating his wort with a yeast plant of known properties. The isolation and testing of such a plant being a time-consuming and laborious process, to say nothing of the amount of apparatus involved, the starting yeast is usually purchased from a laboratory which makes a specialty of such cultures. In ordering, it is only necessary to specify the kind of material to be worked, the concentration, and the average outside temperature. It is best to order the yeast in a cane sugar solution in a sealed two-necked flask.

Ten 250 cc. erlenmeyer flasks are cleaned with a bichromate cleaning solution, rinsed with hot fresh distilled water, and 100 cc. of wort placed in each one. They are then plugged with aseptic cotton, selecting the cotton from a fresh package. These flasks are placed in the air bath at 82° C. and kept at 30° C. after two hours' heating at the higher temperature. After twenty-four hours at 30° C. the heating is repeated. After another twenty-four hours at 30° C. they should show no signs of fermentation. If they do, the process is repeated once more. If no such signs appear after forty-eight hours, they are sterile, and if not sterile after three such heatings it is better to make a fresh start. When the sterile wort is ready, both necks as well as the body of the flasks of starting culture are rapidly gone over with the flame in such a manner as not to heat the yeast unduly. They are then shaken, and the necks are opened by scratching with a file and breaking. A sterilized air filter made from a calcium chlorid tube full of absorbent cotton is attached by means of a sterilized rubber tube to one of the necks and a little yeast poured into each flask in rapid succession, an assistant singeing the cotton, removing, and singeing again, and replacing, before and after each addition. It is well to wash the bench and burners with a 0.5 per cent formaldehyde solution before doing this and to be sure that the floor has not been swept in four hours, the last sweeping being made after sprinkling the floor with the above-mentioned solution. The operators should roll up their sleeves above the elbows and wash the hands and arms with antiseptic soap. These precautions may perhaps seem absurd, but owing to the predominance of predatory bacteria too much care can not be exercised. The 10 flasks are then kept at 30° C. until fermentation has begun. As soon as it is seen that the fermentation has passed its maximum and is beginning to subside the flasks are numbered and a drop transferred from each to a correspondingly numbered microscope slide by means of a platinum loop, using the same precautions as outlined above and heating the loop red hot before each sampling. The slides are then examined and the presence of foreign bacteria ascertained, if present. If any flasks show such, which very rarely happens, they are immediately emptied and well cleaned. Of the remainder two are allowed to ferment at 30° C. until quiescent.

The system about to be outlined dispenses with the so-called "jugs," which have been in use for many years in the plant under discussion. The new system has been in use for one year and has given such general satisfaction that I do not hesitate to recommend it for general adoption. The wort is placed in gallon glass bottles, 3 liters in each. The bottles are then plugged with cotton and sterilized in the same way as the erlenmeyers, with the exception that instead of being heated to 82° C. in an air bath they are brought to that temperature while immersed to their necks in water. After being assured of their sterility, they can be kept indefinitely. Two of the bottles are inoculated from two of the eight unreserved flasks, observing the usual precautions when opening and closing flasks and bottles. They are then allowed to ferment, and it will be very rarely found on examination that one of them is contaminated, and never that both are. If both bottles are good, on the other hand, they can be used on two successive days, when the maximum of fermentation is passed. It is well to keep them at from 25° to 30° C. during fermentation.

Two of the erlenmeyers have been reserved and allowed to become still. These are then shaken and a drop of each poured with the usual precautions into an erlenmeyer sterilized and prepared in the usual manner, but containing 100 cc. of 10 per cent cane sugar instead of wort. These cane-sugar flasks are put away in a cool dark cupboard for use in case of accident or the contamination of the other erlenmeyers, when a fresh start is made precisely as when the culture was first procured. Each time two bottles are started, two erlenmeyers are inoculated first from the same flask. Thus the propagation is made continuous, and the yeast always being in an active state contamination is reduced to a minimum. The erlenmeyers are prepared and sterilized ten at a time and used as needed.

To recapitulate briefly: Having 10 erlenmeyers and 10 flasks sterilized and having sown the culture and reserved two erlenmeyers for the cane sugar, it is necessary each day to set two fresh erlenmeyer flasks from two which are working and to set two bottles from the same flasks. The erlenmeyers which have been set one day are used to set those of the next day, before inoculating the bottles with them. Should the bottles not be required each day, this point must be regulated in accordance with the demand. After an erlenmeyer has fermented, it is good for two weeks for setting purposes.

In the distillery an amount of rye equal to 2.5 per cent of the total grain to be mashed and an amount of malt equal to 2.25 per cent are mashed in about 2.0 liters of water per kilogram of grain. This is mashed at 82° C., adding the rye first and the malt immediately after. After standing four hours at this temperature, this mash is cooled down to 56° C. and kept at from 54° to 56° until the lactic acid reaches a sufficient concentration. At the same time another mash consisting of 0.25 per cent of the total grain used is made in a large tin can in the same manner as for the wort, but without straining off the grain. This mash (called the dona) is then cooled to from 25° to 30° C. and set with a bottle of fermenting wort. In twenty-four hours it is transferred to the big tub, cooled to 20°, in which lactic acid has been developed.

The lactic acid which develops in the tub left at 54° to 56° C. is the result of the activity of organisms always naturally present in such materials. This is the maximum temperature for the growth of these organisms, and their presence, as well as that of the lactic acid, while inhibiting the growth of other bacteria, is nonprejudicial to the yeast. In order to insure such a protective action, the lactic acid must have a definite concentration. It appears that 0.125 normal is the desirable one to use. Should the acid not be of this concentration when the dona is ready, it is necessary to make the mash in advance of the dona, which is considered ready when the concentration has decreased to from 12 to 14 degrees Balling. It is necessary, therefore, to take the Balling of the dona when set and again toward the end of the twenty-four hour period. If it does not work down sufficiently, the fault is that it has become chilled, in which

case the next dona must be kept warmer. If it works too swiftly, the action can be checked by placing the can in cold water.

Having inoculated the main yeast mash with the dona, the yeast immediately begins to act. This mash, which is from 24 to 26 degrees Balling, should be allowed to work down to from 7 to 8 per cent. The temperature should not be allowed to go above 32° C., and when this point is almost reached, the action is checked by running cold water through the coils of the yeast tub. A temperature of over 32° C. soon exhausts the yeast and renders the mash liable to bacterial invasion. Before the temperature has reached 28°· C. in the yeast tub, a can which will hold 5 per cent of the yeast mash is filled. This can, together with a new dona, is used to set the next yeast. After a week this canful of yeast is not used for one day in order to insure that no infected yeast is carried over more than one week, in which time infection can not make much headway. The following statement may serve to make this clearer, in which D represents the dona, S the can taken from the yeast mash, and Y the main yeast mash:

First day: Mash and set D 1 mash Y 1.

Second day: Take off S 1 from Y 1; Y 1 to fermenter; set Y 2 with D 1 and S 1. Mash and set D 2, mash Y 3.

Third day: Take off S 2 from Y 2; Y 2 to fermenter. Set Y 3 with D 2 and S 2. Mash and set D 3. Mash Y 4.

Fourth day: Take off S 3 from Y 3; Y 3 to fermenter. Set Y 4 with D 3 and S 3.

Fifth day: Take off S 4 from Y 4; Y 4 to fermenter. Mash and set D 4. Mash Y 5 and Y 6.

Sixth day: Set Y 5 with D 4 and S 4. Mash and set D 5.

Seventh day: Set Y 6 with D 5; Y 5 to fermenter. Mash and set D 6. Mash Y 7.

Eighth day: Take off S 6 from Y 6; Y 6 to fermenter. Mash and set D 7. Set Y 7 with D 6 and S 6. Mash Y 8.

It is possible to secure very good results without the use of so many donas, leaving them out at times and setting with stock alone. After the still has been running a few weeks this can be tried. In a well-protected yeast room such an addition of fresh yeast in the donas may only be necessary once a week, while in an open yeast room more frequent additions must be made. The purpose of carrying the stock from the yeast tub over is to insure the presence of highly active cells.

The microscopic work incidental to a fuller control of distillery operations is to determine the presence of foreign bacteria, insure the presence of sufficient yeast, and observe its condition. If a still is running constantly, no more can be done, as the chemist's time is too limited to permit of laborious microscopical examinations and these had best be left to the zymotechnical laboratories. It saves much trouble if a large supply of mature and active yeast cells is always in readiness when any particular medium is to be inoculated (bottle, dona, etc.). It will be found that when the dona has worked off to about 14 degrees Balling and the yeast is down to 7 or 8, the desired condition has been fulfilled; hence a count of the yeast in these need only be made at intervals as a check. In the fermenters this condition must be reached before the temperature has risen 15° C., otherwise the danger of bacterial infection is much increased. By starting with an abundance of yeast, this organism is given the predominance and all others are destroyed except the lactic acid bacteria; hence, if the fermenter does not reach the maximum before this time, the yeast mash must be increased enough to meet the demand.

It is only possible, however, for a certain number of yeast cells, about 110,000,000 per cubic centimeter, to live in any given medium at the same time, because if this number is exceeded the source of food supply becomes insufficient and some are starved, so to speak. The number of cells present may be determined by means of a blood slide, which has a cell with the floor so divided that when the cover glass is put on each square forms the base of a cube with a content of $\frac{1}{400}$ cubic millimeter.

The liquid to be examined is diluted 1 to 50 in a 50-cc. flask and a drop is placed on the slide and covered. The slide is kept level for a few minutes to allow the yeast cells to subside, when those on each square are counted. The average of several squares is taken and the total content calculated. The erlenmeyers, bottles, donas, and yeast mash are all examined in this way.

A microscopic examination may also be made to determine the dead cells. This is done in precisely the same fashion except that instead of diluting 1 to 50 with water, a 0.001 per cent solution of methyl violet is used. The dead and exhausted cells take up the stain. Not more than 3 per cent should be present except during the last days of fermentation. The usual cause of an excess of these is the use of too high temperatures. The presence of foreign bacteria in the donas need cause no alarm, but under no circumstances must they be present in the erlenmeyers or bottles. After the yeast has been propagated in the distillery the lactic acid protects it.

In conclusion it must be insisted on that some regular schedule be adopted and maintained or good results can not be expected. You can not set yeast on Monday and use it Friday. It is not expected that the process here given will be followed exactly, nor has it been described with the minuteness necessary for an exact reproduction, but the procedure has been briefly sketched to serve as a basis for other schemes adapted to each particular plant. The feature of dispensing with the use of copper yeast cans and jugs will be found very desirable for small plants on account of the greater economy.

PRACTICAL OPERATION OF A DISTILLERY.[a]

By R. P. O'BRIEN, *Freeport, Pa.*

In the location of a distillery the most important questions from a practical standpoint are the water supply, facilities for shipment, and environment in general. You may find in the text books information regarding the kind of water best suited to distilling operations, but they are usually silent as to the amount of water that is required to operate a distillery of a given capacity, whereas it is essential to know both. In practice I find that to operate a distilling unit of 1,000 bushels per ten hours the minimum water supply must be 250 gallons per minute.

The shipping facilities for receiving the raw material and disposing of the finished product are important also. The best location for a distillery would naturally be in the midst of the grain fields that produce the raw material provided there is available a sufficient quantity of the right kind of water and also means of shipping the finished product.

The next question is the capacity of the plant and the requisites as to building and machinery for the particular capacity adopted. For example, a unit of 1,000 bushels per ten hours will require about 600 horsepower boiler capacity and 300 horsepower engine capacity to be accompanied by a mill for grinding 1,000 bushels in ten hours. (A six-brake corrugated roller mill 9 by 34 will furnish that capacity.) The still to be required would be 10 feet in diameter by about 28 feet high, separated into chambers and furnished with pressure gauges and thermometers on each chamber, together with the other ordinary equipment, connected with a doubler about 4 feet in diameter of approximately the same height as the still and having a condensing worm of 1,250 square feet. The mashing apparatus should consist of two mash tubs each 16 feet in diameter and 5 feet deep, provided with three-arm mixing rakes, the

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[a] While Mr. O'Brien's lecture describes the conditions and operations in a distillery for making potable spirits, the fundamental principles of fermentation are the same and directly applicable to the process of making industrial alcohol from grain, and the estimates as to capacity and equipment are of special interest.

necessary thermometers, etc. The yeast tubs should be 7 feet in diameter and 6 feet deep. A cooling worm for this mashing capacity should consist of about 2,000 feet of incased 3-inch copper pipe, and it would require nine fermenters, each with a capacity of 333⅓ bushels at 45 gallons per bushel, and two receiving tubs each of about 5,000 gallons capacity, together with the necessary pumps, scales, and conveying machinery essential for handling the material. The essential working tools of the operatives must be provided, which would consist of thermometers, saccharometers, hydrometers, acidimeter and iodin test apparatus, and a microscope, also the usual accessories for examining yeast for contamination.

The grain is the next factor to receive our attention. Various grains are used in the manufacture of alcohol, but corn is used most extensively, and it may be either malted or unmalted. In all distilling operations a certain percentage of the grain must be malted in order to effect hydrolysis of the starch in the unmalted grain. In the manufacture of alcohol it is not necessary to prepare the grain so carefully, because the high heats used in the mashing process inhibit contamination resulting from dirty or inferior material. A portion of the grain is prepared by the malting process, which consists of steeping it in tanks a certain length of time until it absorbs sufficient water to properly start the germination and after this is accomplished remove it from the steeping tanks to the growing floors or to drums, as the case may be, and there grow it until such time as it will have reached its highest diastatic value, then place it in the kiln, thereby arresting further growth and also drying the grains, maintaining such a heat in this process as will not injure its diastatic value and at the same time dry it sufficiently to stand storage.

The raw grain and the malt having been prepared, we are ready to begin distilling operations. Both the malt and grain are usually brought on to the distillery premises and into the mill, where they are ground by means of a gradual reduction mill, and the resultant meal is then placed in weighing hoppers directly over the mashing tuns. Then place in the mashing tun about 25 gallons of sterile water for every 56 pounds of meal in the scale hoppers above the mash tuns. The temperature of the water at the beginning of these operations should not exceed 140° F. The raw grain is first let into the mash tun and thoroughly mixed with the water; then the mashing temperature is raised to about 162° F. in the case of rye and 212° in the case of corn. In alcohol distilleries in lieu of mash tuns an inclosed cylindrical cooker may be used in which the temperature is raised to upward of 300° at this period of the mashing. These cookers are to be provided with at least three thermometers, a steam-pressure gauge, a vacuum gauge, and a tap cock on the side for testing conversions, and also with a vacuum pump for lowering the temperature before adding the malt to the mash. In this particular the alcohol process differs from that for making potable spirits.

After the mash of the raw grain has attained the required heat of not over 162° F. it is allowed to stand long enough for the proper cooking of all the particles of raw grain, the time varying with the coarseness of the meal. After it is sufficiently cooked, the mash is cooled to approximately 155° F. and enough malt for diastatic purposes is added. Hydrolysis will take place in from twenty minutes to two hours, according to the quality of the materials used. During this period the temperature should be uniform, never below 145° F. nor above 158°. Before cooling this mash for fermentation the iodin test should be applied to see if the conversion is complete, after which the mash should be cooled as rapidly as possible and placed in the fermenter. Preferably, inclosed pipes should be used for cooling, as the mash should never be exposed to the air when at a temperature that would encourage foreign fermentation. At this time the yeast for this mash should be in the fermenter, so that fermentation can start immediately upon the arrival of the mash.

The yeast for this mash should be prepared of equal parts of unmalted rye and malted barley and the grain should constitute about 4 or 5 per cent of the mash to be fermented. It should be scalded in much the same manner as the mash and allowed to stand forty-eight hours to undergo a lactic fermentation, which should be conducted by pure culture methods, the lactic germs being destroyed in the yeast mash before setting it with the proper yeast germ. The specific gravity of the yeast should be anywhere from 25 to 30 degrees Balling and should work down to from 8 to 12 degrees Balling before it is used to ferment the mash. The exact figures for these details are largely a matter of opinion among different distillers, and to be exact would possibly invite criticism, but experience will soon teach each individual distiller what procedure is best suited to his conditions. The figures given are a reasonably safe guide as to limits.

After the fermentation is complete the beer should be tested as to its degree of attenuation, acidity, and starch conversion as a check upon the mashing operation. If these determinations are satisfactory, the beer is sent to the still, which should be properly charged and started in operation, care being taken not to overload, so as to cause frothing. The distillation should be conducted at as low a temperature as is consistent with speed and the kind of spirits desired, and as the condensed spirits flow into the separator the distiller can determine the strengths of the distillate and govern the division accordingly, returning the undesired portion for redistillation and sending the satisfactory spirits to the tanks for reduction and bonding.

It should be the idea of every distiller to construct his distillery so that every portion of it is easy of access for cleaning and repairs. Everything should be accessible for proper sterilization by steam, all apparatus being connected with live steam pipes and thoroughly sterilized when not in operation. Cleanliness is absolutely essential to the proper operation of a distillery, and no distillery can be successfully operated if this injunction is disregarded.

EFFECT OF DISTILLATION IN DIFFERENT TYPES OF STILLS.

By L. M. TOLMAN, *Bureau of Chemistry.*

The object of distillation is the separation of a volatile liquid from a nonvolatile substance, or more frequently the separation of two or more liquids of different volatility. The latter form of distillation takes place in the preparation of alcoholic distilled spirits, the more volatile alcohol being separated from the water. While this separation of alcohol and water is the chief end in view in the distillation of various fermented liquids, we know that the distillate obtained from a fermented mash of any kind is not simply alcohol and water, but contains a number of other substances, present either as impurities due to the fermentation or as natural constituents of the material used in making the mash.

It is only necessary in making alcohol for denaturing purposes that it should be of high alcoholic strength, but in order to have an intelligent comprehension of the separations taking place in the various kinds of stills it is necessary for us to have some idea of what takes place in fractional distillation. The object of fractional distillation is the separation from each other of the various substances having different boiling points, and ordinarily this is accomplished by keeping the distillates in separate portions and then redistilling them. After repeated distillation certain portions may be obtained which are almost pure substances. It is practically impossible to obtain a pure ethyl alcohol by redistillation in a simple still, but by repeated distillations the alcoholic strength may be sufficiently increased for denaturing purposes. In practice, however, other devices have been adopted which will give a very pure,

strong alcohol in one operation. This is accomplished in what is known as the "column still."

In the column still are a great number of small boiling chambers, which are partly filled with condensed vapor from the chamber below, and are also boiled by the vapors from that chamber. In the top chambers will be found an almost pure ethyl alcohol containing about 5 or 6 per cent of water.

The difference in composition between the different portions of the distillate from a simple still and those from the column still is readily shown by the following experiments. The first was made on the distillate from a pot still. An alcohol solution was prepared, approximately 50 per cent by volume, to which were added various constituents ordinarily occurring in a distilled spirit of this strength. Of this 10 liters were placed in a copper still and the distillation started. Each 500 cc of the distillate was kept separate and an analysis made, the results of which are given in the following table:

Effect of pot still distillation.

[Parts per 100,000.]

No.	Portions of distillate, sample, and residue.	Proof.	Acids.	Es-ters.	Alde-hydes.	Fur-fural.	Fusel oil.
	cc.	Degrees.					
1	1st 500	162.5	9.6	462.0	400.0	2.5	94.1
2	2d 500	162.6	9.6	315.9	240.0	2.0	104.7
3	3d 500	160.8	10.8	....	156.0	3.0	109.1
4	4th 500	159.9	10.8	116.5	75.0	4.5	119.7
5	5th 500	158.0	12.0	58.2	40.0	6.5	145.2
6	6th 500	156.1	14.4	27.3	17.0	8.0	176.8
7	7th 500	152.8	18.0	10.0	7.8	8.5	176.9
8	8th 500	148.9	20.7	3.6	3.6	9.5	188.3
9	9th 500	142.7	25.2	3.6	2.4	14.5	179.5
10	10th 500	131.3	34.8	5.4	2.6	17.0	149.6
11	11th 500	110.6	48.0	5.4	6.6	14.0	82.7
12	12th 500	73.3	76.8	10.9	5.6	....	42.2
13	13th 500	31.1	96.4	11.8	6.4	17.0	9.7
14	14th 500	6.7	130.8	11.3	6.6	45.0	5.3
15	15th 500	2.7	147.3	11.8	3.6	7.0	2.6
16	16th 500	2.2	159.1	14.5	18.0	.0	.0
17	Original sample	92.9	93.6	78.3	64.0	8.5	....
18	Residue	.0	239.4	34.6	4.6	.0	None.

These results have been platted in fig. 14, and show a very interesting condition. The alcoholic strength of the first 500 cc. is a little above 80 per cent by volume, and a very gradual decrease in strength takes place until about half of the original volume has been distilled over, when the alcoholic strength drops very rapidly. The esters, however, come over chiefly in the first part of the distillate, very rapidly decreasing until there are practically none in the eighth fraction, which shows a very remarkable separation of ethyl alcohol from the ethyl acetate, in spite of the fact that their boiling points are almost identical, as will be seen when it is noted that the alcoholic strength of the eighth fraction is still between 70 to 80 per cent by volume, and yet is almost free from esters. It will also be noted that the aldehydes follow a course very similiar to that of the esters, and on account of their very low boiling point this might be expected. With the acids, however, a different condition of affairs appears. The successive fractions show a gradual increase in acidity and the residue, after the alcohol has been distilled off, still shows a considerable amount of acid.

In the case of the fusel oil quite a different series of changes takes place. The fusel-oil content of the distillate gradually increases until the eighth fraction has come over, and then gradually decreases with the alcoholic strength of the distillate, dropping down to practically zero when the ethyl alcohol is entirely removed. An analysis of the residue does not show even a trace of fusel oil, indicating that this high boiling point alcohol completely passes over in a distillation of this kind; this has been the general experience in our analytical work and therefore a simple distillation is de-

pended upon in the determination of fusel oil to separate it from the extractive mat-
ters and other substances which might interfere with its estimation. This condition
in the distillation of fusel oil is not like that obtained when products are carried over
mechanically by means of the steam, but is undoubtedly due to the solubility of the
vapors of the fusel oil in those of the ethyl alcohol.
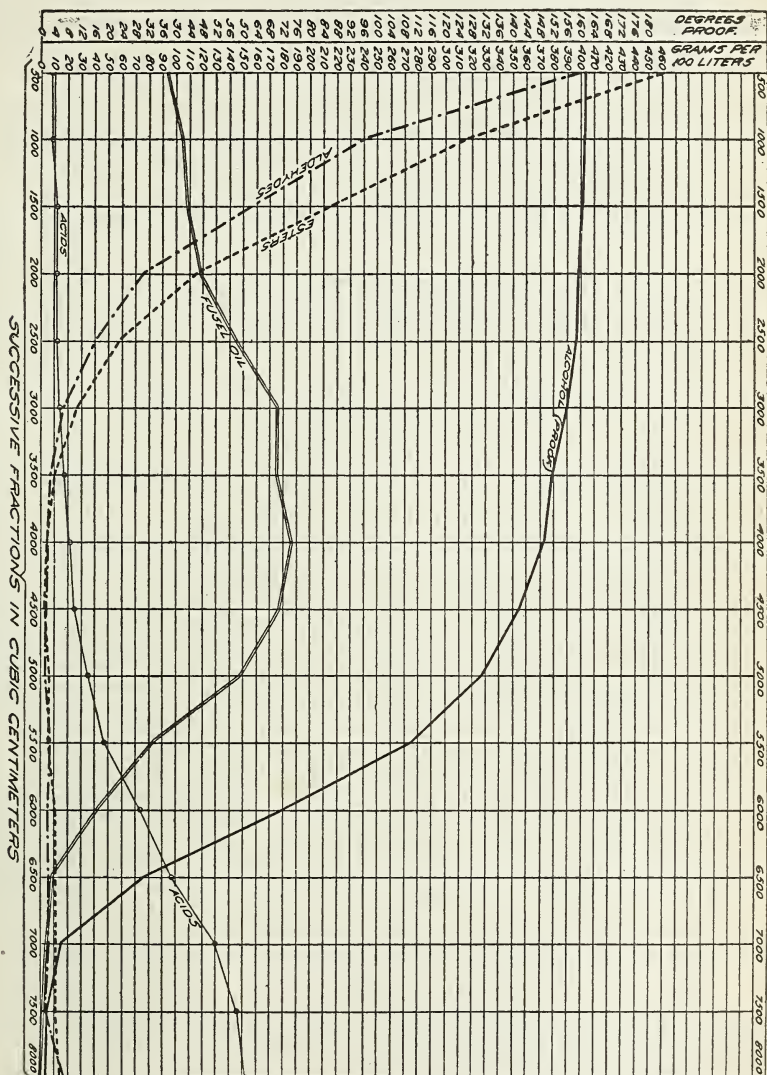
Fig. 14.—Fractionation in a pot still.

The furfural behaves like the fusel oil in that there is a gradual increase in the con-
tent of furfural as the distillation goes on until a certain point is reached, when it be-
gins to decrease, and in the last distillate, which is free from alcohol, there was no
furfural found, indicating that it had been entirely distilled over. This is also in har-
mony with the analytical methods employed in the past which use a distillate pre-
pared by simple distillation for the determination of furfural. These results are in-

teresting in showing what amount of fractionation takes place in simple distillation, and they also show that when all of the fractions which contain appreciable amounts of alcohol are mixed together the resultant liquid will be of practically the same composition as the original solution with the exception that there will be a small loss of acid. This is also in strict accordance with our experience in the analysis of distilled spirits, which indicates that all of the aldehydes, esters, furfurals, and fusel oils will be found in the distillate prepared by distilling from a small flask, so that the result
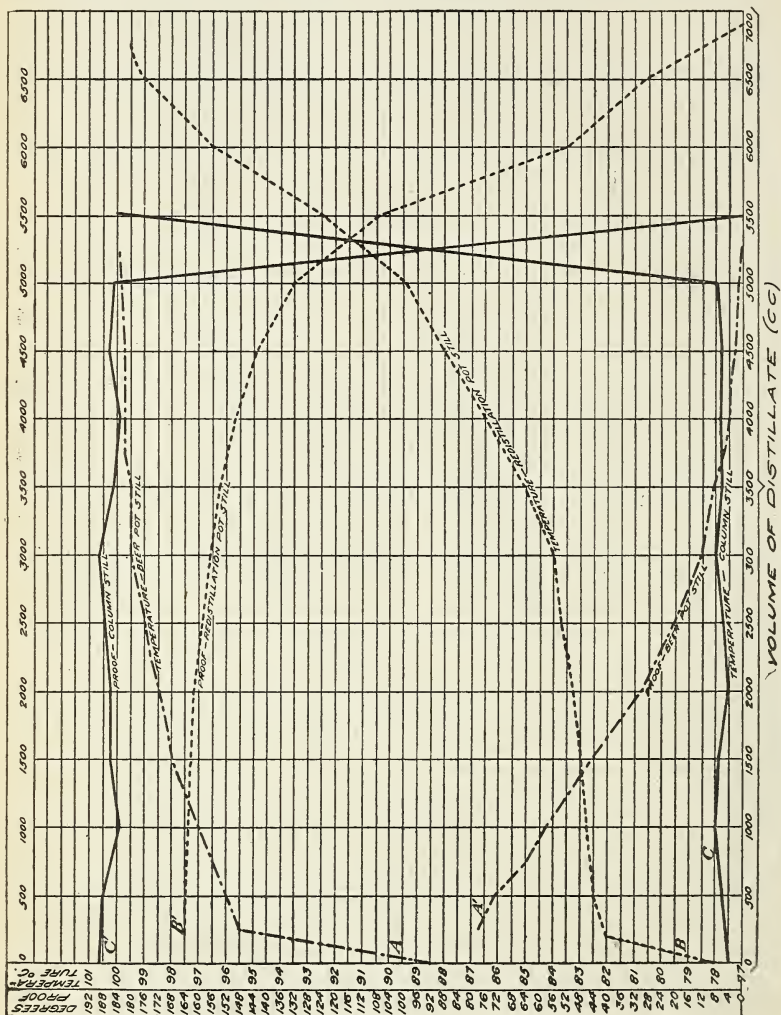
FIG. 15.—Comparison of proof of distillate and temperature in column and in pot stills.

of the analysis of the distillate is satisfactory. We have also found, however, that in the case of the acids only a part will be found in the distillate, so that it is necessary to make the determination of acid upon the original material.

In connection with these results, attention is called to the chart (fig. 15) which shows the difference in temperature of the vapor at various stages of the distillation in a simple still and in a still with a rectifying column. In this chart, the line A shows the changes in the temperature of the vapor in a beer still as the distillation

takes place. It increases very rapidly from the temperature of approximately the boiling point of alcohol to 95° C., and then gradually increases until it reaches the boiling point of water. The line A′ shows the curve of the percentage of alcohol in the distillate, which is of course practically the reverse of the temperature curve, starting at about 40 per cent alcohol and rapidly decreasing to zero. Line B represents the temperature of the vapor in a pot still during the distillation of an alcoholic solution at approximately 50 per cent by volume of alcohol which compares with a second distillation in a simple still, while B′ represents the percentage of alcohol in the various portions of the distillate. Line C shows the temperature registered in the top chamber of the column still, while C′ represents the proof of the distillates during the same period. It will be seen from this chart that the temperature remains very uniformly at approximately the boiling point of ethyl alcohol until practically all of the alcohol has been removed, when the temperature rapidly rises to the boiling point of water, while the percentage of alcohol rapidly drops to zero. The curves in this chart show what a difference in composition there must be in the distillates represented by lines A′, B′, and C′.

In order to demonstrate still further the difference between the results obtained in the pot still and in the column still, attention is called to the following table, which gives the analytical results on fractions of the distillate prepared in a column still:

Analysis of various fractions obtained in a column still.

[Parts per 100,000 referred to 100° proof.]

Determination.	Successive fractions of distillate. (cc.)				
	1st 500.	2d 500.	3d 2,350.	4th 1,150.	5th 500.
Acids	0.9	1.3	2.3	1.3	2.5
Esters	1.3	.0	Tr.	.0	.0
Aldehydes	13.4	1.3	2.5	1.1	.6
Furfural	.0	.0	.0	.0	.0
Fusel oil	.0	19.0	19.0	35.0	500.0
Proof	186.4	184.5	184.6	186.2	123.3

It will be seen that the separations in the column are quite different from those in the pot still; especially is this true regarding the fusel oil, which in the first fraction is practically present only in traces and no appreciable amount is noticed until toward the end of the distillation. Finally when the percentage of alcohol in the distillate begins to decrease, the percentage of fusel oil greatly increases and at the end a product is distilled which is practically pure fusel oil—quite a different effect from that shown in the pot still, where the fusel oil comes over with a high percentage of alcohol, decreases in amount as the percentage of alcohol decreases, and is all distilled before the last trace of ethyl alcohol comes over.

This experiment does not afford an entirely satisfactory comparison as the material distilled in this case was purified before distillation by means of caustic potash and nitrate of silver, the first to remove the acids and esters, and the second to remove the aldehydes. If this had not been done, it could have been shown, as later experiments demonstrated, that the first portion of the distillate coming over from the column still is very high in esters and aldehydes, and in this respect is somewhat similar to the first distillate from a pot still, though less rich in these constituents. As will be seen, there is a considerable fractionation taking place in a pot still and various portions of the distillate show quite a difference in composition. If these various portions were kept separate and redistilled, a fair separation of their con-

stituents could be effected in the course of several distillations, but in the ordinary distillation in a small still, where the distillates are combined, the final distillate will have practically the same composition as the original solution, except for a concentration of the alcohol and a reduction in acid. On the other hand, in the column there will be almost a complete separation of the aldehydes and esters in the first fraction, a comparatively pure neutral alcohol in the middle portion of the run, and the latter portions will gradually become more impure, containing large amounts of fusel oil, until in the tails of the run quite large amounts of fusel oil are found and there will be an actual separation of pure fusel oil at the end.

For the reasons previously stated, the results in the last table do not show as well as they might the fractionation taking place in a column still. The following table was therefore prepared, giving the results of analyses of fractions prepared in a laboratory column still from a spirit which had matured for several years and had not been treated in any way. It shows, therefore, much more accurately the separations taking place.

Analysis of fractions in a column still.

[Parts per 100,000, 100°-proof alcohol.]

Successive fractions.	Proof.	Acids.	Esters.	Alde-hydes.	Fur-fural.	Fusel oil.	Total distillates and residue.[a]
	Degrees.						cc.
First portion	188.6	1.5	158.0	19.1	None.	Trace.	4,300
Second portion	187.4	.9	42.3	6.0	...do..	...do..	5,600
Third portion, or middle	189.2	.9	2.7	1.2	...do..	...do..	20,000
Fourth portion, or last of middle	187.2	.8	4.2	1.4	...do..	33.4	8,000
Fifth portion, or tails	42.0	18.0	49.3		...do..	1,065.0	6,500
Sixth portion, or fusel oil							200
Residue in still	None.	65.4	11.4	None.	None.	None.	40,400

a Sum total 85,000 cc.

These results show that the esters and aldehydes come over in the first portions; that the middle run is practically free from everything except alcohol; that the fusel oil begins to come over in the last of the middle run, and that the tails still contain quite a large amount of this constituent. In this distillation which, as before stated, was made in the small laboratory column, about 200 cc. were recovered as fusel oil in the sixth fraction. The residue left in the still contained a small amount of acid. It is also worthy of note that there are esters in the tailings, indicating that some of the esters of the higher alcohols are present.

The following set of samples taken at a large alcohol distillery show the changes taking place on a commercial scale:

Analysis of fractions from a large alcohol distillery.

[Results calculated to 100° proof in parts per 100,000.]

I. S. No.	Substance.	Proof.	Acids.	Esters.	Alde-hydes.	Fur-fural.	Fusel oil.
5294–a	High wines	128.9	42.0	19.7	2.5	0.0	102.0
5295–a	Rectified spirits	108.5	7.7	Tr.	4.1	.0	93.0
5297–a	No. 2 spirits	190.8	2.8	6.4	1.6	.0	Tr.
5298–a	No. 1 spirits	190.3	1.1	Tr.	.6	.0	Tr.
5299–a	Middle run or velvet spirit	192.2	1.3	.0	.6	.0	Tr.
5300–a	Alcohol	190.7	3.4	14.7	3.4	.0	Tr.
5296–a	Low wines	170.6	2.8	39.3	21.1	.0	a1,634.0

a 1.63 per cent.

Description of samples.

5294–a. This is an average sample of the spirit high wines, produced by their beer still, drawn from the receiving cistern, when distillate was about to be reduced in proof, prior to passing through charcoal.

5295–a. This sample represents spirit high wines after dilution and passing through charcoal leaches. The following samples are obtained by fractional distillation of this material.

5297–a. Mixture of third and seventh fractions obtained in redistillation of rectified spirits in alcohol column.

5298–a. Mixture of fourth and sixth fractions obtained by redistilling rectified spirits in alcohol column.

5299–a. Mixture of fifth fraction obtained by redistillation of rectified spirit in alcohol column.

5300–a. Mixture of second and eighth fractions obtained by redistilling rectified spirits in alcohol column.

5296–a. First runnings and tailings mixed (average sample) in redistilling rectified spirit in alcohol still.

All of these results show very clearly the difference in the separation made in a single distillation as compared with that taking place in a modern column still.

BACTERIOLOGICAL TECHNIQUE.

By Geo. W. Stiles, *Bureau of Chemistry.*

Pasteur was the first investigator to reveal the phenomena of fermentation. In 1862 he published a paper entitled "On the Organized Corpuscles Existing in the Atmosphere." His work among the vineyards of France possesses the greatest value to the manufacturer of alcoholic beverages, and particularly so to those engaged in the production of denatured alcohol.

One of the greatest difficulties to-day in bacteriological work is the lack of trained men with laboratory experience. This can not be acquired in a short time, as the technique is attained only by constant application and strict attention to details. The following fundamental facts of which the bacteriologist must make use, while elementary, are submitted as of possible convenience and interest in connection with the incidental bacteriological work done in a distillery, although this is for the most part left to special laboratories.

Definition of bacteria.—Bacteria are minute, unicellular organisms, generally recognized as belonging to the vegetable kingdom. Contrary to the general belief, they differ from such organisms as the *Plasmodium malaria*, the causative agent of malaria, which belongs to the animal kingdom.

Classification of bacteria.—Various schemes have been suggested for the classification of bacteria, but the commonly accepted method is based upon a combination of morphological and biological characteristics, three general types being recognized:

(1) Micrococci—generally spherical in form; grouped singly, in pairs, clusters, chains, fours, cubical packets, etc.

(2) Bacilli and bacteria are rod-shaped organisms, either motile or nonmotile, the kind of movement depending upon the number and distribution of their flagelli. They may be of various sizes, either straight or slightly curved, chain-forming or single, with ends rounded or square, etc., or, again, they may or may not possess spores (which correspond to seeds of higher plants), or they may assume involution forms under abnormal conditions. Some organisms like *B. diphtheriæ* show a remarkable variety of forms, and vary somewhat according to the kind of media upon which they grow, the temperature and other factors concerned in their development.

(3) Spirilla are corkscrew-shaped rods exemplified by the spirillum of Asiatic cholera. This type of organism is not so frequently encountered as the bacilli or micrococci groups.

Culture media.—The basis of most ordinary artificial culture media is peptonized beef broth. This may be prepared from lean beef or beef extract. Other substances used wholly or in part for culture media purposes are potato, bread, milk, agar, gelatin, wort, blood serum, ascitic fluid, egg, sugar solutions, various decoctions, fruit juices, etc., according to the nature of the organisms to be grown. In addition to these we regularly use Dunham's peptone solution and Dunham's rosolic acid medium and nitrate broth. It is not necessary to give a detailed description of the preparation of these various media, since all of the modern works on the subject give the theoretical side of their preparation; but in order to gain practical knowledge it is essential to go to the laboratory for experience. A few suggestions, however, may be of value. Accuracy in bacteriology is essential. One must work precisely and be able to grasp a difficult problem with a clear understanding of the necessary requirements. The greatest care must be taken to insure that all culture media are absolutely sterile before being used, and to provide against such contamination a good practice is to incubate all media in a warm room two or three days before use; gelatin, however, might be excepted. Much time may be saved by making a large quantity of the culture medium and keeping it after sterilization in flasks ready for distribution into test tubes. It is desirable to keep tested stock culture media in the ice box to prevent undue desiccation and concentration.

Sterilization of culture media.—Certain media may be obtained free from contaminating organisms, as ascitic fluid, for example, although in most instances it is necessary to employ some degree of heat to render the product sterile and in a proper condition for use without danger of foreign contamination. Fractional or intermittent sterilization may be employed for this purpose, or a single continuous heating in the autoclave under pressure for a short period may bring about the same results.

Some media, such as gelatin, sugar solutions, blood serum, milk, etc., are very susceptible to high temperatures, being quite readily altered in character; these are best sterilized by the intermittent method, that is, thirty minutes a day at boiling temperature for three successive days, leaving the media at room temperature during the interval to permit nonvegetative forms to develop, thus rendering them more sensitive to the action of heat. Spores are difficult to sterilize and may withstand continuous heating at ordinary boiling temperature for hours. They are, however, generally killed in the autoclave under one or two atmospheres pressure for from twenty to thirty minutes' exposure.

Various methods are in use to make plugs for glassware, test tubes, flasks, etc. They may be made by rolling thin layers of either absorbent or plain cotton into elongated masses, binding in the fringed edges, or by the use of a rod inserting a small pledget in the mouth of the tube or receptacle to be stoppered. Plugs should fit snugly, but not be too tight.

Absolute sterility must be observed with glassware as with culture media and all other material except the specimens under consideration. The final reaction of ordinary culture media should be made as uniform as possible, and the recommendations made by the American Public Health Association[a] in regard to culture media should be observed.

Dangers from fire may be lessened by making connections with burners, thermostats, etc., with metal instead of ordinary rubber tubing. The floor of the laboratory should be daily scrubbed or wiped with some mild germicidal agent, 5 per cent carbolic acid or other effective solution, to prevent unnecessary prevalence of germ life in the

[a] Report of committee on standard methods of water analysis to the laboratory section of the American Public Health Association, page 104. Reprinted from Journal of Infectious Diseases, Supplement No. 1, May, 1905, Chicago.

working room. Tops of tables may also be wiped with such a solution with great advantage. Infected material of any kind should be cared for at once, and old discarded cultures should be boiled and not allowed to accumulate around the laboratory. In fact, one must pay the closest attention to each little detail, neglecting no part of the work, and the best results are only obtained by systematically keeping each part of the operations up to date.

Currents of air are likely to contaminate and render valueless what might otherwise be a careful piece of work, thus one should be provided with a small well-lighted room, where plating and inoculations can be made with the least opportunity for contamination.

One should prepare check cultures whenever it is possible, and also expose agar plates about the workroom for various lengths of time in order to ascertain the bacterial condition of the air and thus be able to guard more intelligently against infection of the cultures by organisms.

Cleaning and sterilization of glassware.—The proper cleaning of glassware and its preparation for use in the bacteriological laboratory is no small matter, and more care should be given to this phase of the work than is generally given. Petri plates should not only be thoroughly cleaned, but well matched before sterilization. Various soap mixtures and an unlimited supply of hot water must often be supplemented by the bichromate-sulphuric acid mixture to remove stains and cloudiness from glassware which otherwise becomes almost useless. The best grade of Jena or Non Sol glassware may be used, although they contain zinc and boracic acid in their composition, which might tend to inhibit bacterial growth; on the other hand the ordinary soft glass is easily etched and some practical tests have shown an increased amount of alkaline substances present in tubes after boiling in distilled water; this condition may or may not influence the growth of bacteria, yet it is a point worthy of consideration and its importance remains to be determined by the bacteriologist.

Glassware should be sterilized in a dry air sterilizer for at least one hour at a temperature of from 160° C. to 170° C. An ordinary baking oven may be utilized for this purpose by heating a sufficient time to slightly brown a pledget of cotton in case a thermometer is not available.

Keeping of records.—It is of the greatest importance to keep the records in such a manner as to be readily available. Card catalogue systems and properly arranged serial number books prove of inestimable value. Exact notations should be made at the time of making an experiment or immediately afterward.

Microscopes.—The best compound instruments are desirable and for the most exact work the apochromatic objectives should be selected. Oil immersion lenses are not always essential in examining organisms the size of yeast cells, but smaller germs can not be seen to advantage without such a lens. Great care should be observed in caring for a microscope, since its efficiency may be quickly impaired by careless handling. The beginner should be especially cautious in the use of the oil immersion lens, taking care not to force the objective against the glass slide holding an object to be examined. Lens paper should be used to remove cedar oil after the use of the instrument, alcohol or xylol never being used for this purpose. These instruments should be protected from chemical fumes and from dust by being kept in their cases and not allowed to remain standing on the desk for unnecessary periods of time. A northern light is generally preferable for microscopic work when artificial light is not utilized. One should learn to use both eyes interchangeably without being compelled to keep the unemployed eye closed during observations.

It is often advisable to prepare cultures in duplicate or even triplicate on various media, and incubate them at different temperatures. Ordinary air-borne germs grow best at room temperature, while others flourish at a lower or higher temperature. We must determine by experiment and observation the optimum conditions for the growth of the germ that is to be used and supply them.

MICROSCOPICAL EXAMINATIONS.

By B. J. Howard, *Bureau of Chemistry*.

Besides its use in the control of the yeasts and bacteria, the microscope is employed principally in the manufacture of alcohol for the examination of the starches. The most important starch-containing materials to be examined are the cereals (corn, barley, rye, wheat, and rice), tubers (potatoes), roots (cassava and sweet potatoes), and fruits (banana). The characteristic appearance of these bodies under the microscope is briefly described for convenient reference in this connection.

Action of starch toward polarized light.—There is no point in the microscopical examination of starches on which authors disagree more than concerning this property. Some writers record certain materials as inactive, while others credit the same as being active. Our experience with several micropolarizers shows that they vary considerably as to their effectiveness and it seems probable that this may account, in part at least, for the variations recorded.

The starches vary widely in their activity. As a general rule, the larger the grains the greater the activity. Thus, a small grain of potato starch is less active than a larger grain. Besides this, the specific activity of the different varieties must be considered, since a starch grain of wheat, though of greater diameter than a given potato starch grain, may yet be much less active.

Cornstarch.—The grains of this starch are normally polygonal in outline, having four to seven angles. In addition to these, which are the most characteristic forms, nearly spherical grains are not infrequently found, especially in the mealy parts, while the angular forms are met with in the flinty portions. The hilum is located centrally in the starch grains and varies in form from a dot, or short fissure, to a cross or star-like form. The rings are rarely visible. With polarized light a strong central cross is obtained and strong colors with the selenite plate. The size varies from 4μ to 30μ, most of the grains being about 15μ in diameter.

Barley starch.—This starch is composed of lenticular grains varying in size from 2μ or 3μ for the smallest grains to 35μ, most of the grains being between 20μ and 30μ. Rings are indistinctly seen on some of the normal grains. The hilum, which is centrally located, is almost always invisible, but when seen is usually in the form of a faint dot, rarely a slit or cross. Barley starch is only moderately active toward polarized light and gives medium low colors with the selenite plate.

Wheat starch.—This is very similar to the barley starch but the grains are somewhat larger (up to about 50μ), though occasionally small angular grains occur. The rings, hilum, and behavior toward polarized light are virtually the same as noted under barley starch.

Rye starch.—This starch is lenticular in form with grains reaching as high as 55μ. The hilum, which is central, frequently appears in rye as a cross or star, and this may be used to advantage in distinguishing it from wheat or barley. The rings and behavior toward polarized light are practically the same as in the case of barley.

Rice starch.—The grains of this starch are polygonal in form, and in size average about 5μ (with a minimum and maximum of from 3μ to 11μ) diameter. The rings are invisible under ordinary conditions. The hilum, when visible, appears as a central dot. With polarized light faint central crosses are observed, while with the selenite plate a slight play of color is produced.

Potato starch.—This is one of the largest of the starches, being as large as 100μ in diameter, though most of the grains are considerably smaller (60μ to 70μ). They are shaped like an oyster shell, with the hilum usually located near the smaller end, and clearly marked, as a rule. The rings are very prominent and eccentric in position. The grains are very active toward polarized light, forming crosses with the bars passing through the hilum. With the selenite plate a play of high colors occurs.

Cassava starch.—A large proportion of the starch grains of this plant occurs attached to one or more other grains. In the commercial product the grains are mostly separated from each other, appearing as the sugar-loaf type of grain, with one or two truncated surfaces (very rarely more than three), so common in this variety. The grains average about 15μ and rarely exceed 30μ for the largest grains. The hilum is usually very prominent, consisting of a dot, slit, cross, or star. The rings are fairly clear. Strong activity is observed toward polarized light and good play of high colors with the selenite plate.

Sweet potato starch.—This is obtained from the roots of the sweet potato (*Batatas edulis* Clois., *Ipomœa batatas* Lam.). The grains are of the sugar-loaf shape similar to the cassava, but commonly having more truncated surfaces, not infrequently reaching five or six. The grains occasionally are 55μ in diameter, and grains 35μ to 40μ are quite common. Some of the rings have a considerable eccentricity and they are medium strong. The hilum is of the dot, slit, or star type and very prominent. With polarized light the grains show different degrees of activity, some grains being very active while others are scarcely medium in strength. With the selenite plate some show high colors, others low shades.

Banana starch.—This starch is interesting because of its irregular, varied forms. Some of the grains are oyster-shell shaped, others ovoid, others long and slender, and still others curved. They vary in size up to 100μ for the maximum length. The rings are very distinct and the hilum strongly eccentric. The grains react medium strongly with polarized light, the cross bars being very irregularly defined. With the selenite plate a play of low colors is produced.

Action of heat on starch.—Starch heated in the absence of much moisture is converted more or less completely into dextrin. A sample of dextrinized starch mounted in alcohol may appear not much different from the normal starch, but when mounted in water the grains swell more or less and lose their characteristic features according to the extent to which the dextrinization has been carried.

With larger amounts of water the starches become gelatinous. In potato starch the first indication of a change taking place as the temperature is raised occurs at about 60° to 63° C. At this stage there is developed a V-shaped marking of granular or line-like appearance, one arm extending backward each way from the hilum. The first actual swelling begins at about 64° to 68° C. and ends at 73° to 79° C. The rather interesting point was observed, while making these determinations, that individual specimens of potatoes vary somewhat as to the temperature at which these changes take place. In the following table are given the results of some experiments made by the Microchemical Laboratory upon the gelatinization point of some common starches:

Gelatinization point of starches.

Starch.	Swelling begins.	Every grain swollen.	Starch.	Swelling begins.	Every grain swollen.
	°C.	°C.		°C.	°C.
Potato No. 1...................	64	73.5	Wheat (common).............	58	63
Potato No. 2...................	66.5	78	Wheat (durum)	55	63
Corn.............................	65	71.5	Rye.............................	54.5	64
Sweet potato No. 1............	68	79	Cassava.......................	58.5	73
Sweet potato No. 2............	65	78	Banana........................	69	73.5

Toward polarized light, the first part of the starch grain to lose its activity is the center, the effect gradually extending to the periphery.

In the preparation of mash from potatoes or similar materials, the starch alone is not the only portion to be considered. In this case the starch is cooked within the cells and the middle lamella is so softened that the cells readily separate one from the other.

This results in softening the cell walls sufficiently so that during the mashing process they are broken and the starchy contents liberated. The mashing was continued in the samples examined in the Bureau of Chemistry until the swollen grains of starch had been thoroughly broken up and become well mixed with the water so as to form a paste very nearly homogeneous. There is some difference between the starches as regards the manner of swelling, some becoming very irregular and others forming spheres with a clear center so that when viewed under the microscope the grains appear as faint rings with almost perfectly smooth, regular outlines.

SOME SPECIAL USES OF INDUSTRIAL ALCOHOL.

USE OF ALCOHOL IN VARNISH MAKING.

By F. P. Veitch, *Bureau of Chemistry.*

Among the various uses to which alcohols may be put is that of the preparation of spirit and pyroxylin varnishes, lacquers, and enamels. It is not proposed to go into the detail of the manufacture of these materials, the number of which is legion. It will be sufficient for our purpose to consider typical formulæ and processes of manufacture of the chief classes, that a general knowledge of the possible markets for alcohols in these industries may be had.

The term "varnish" embraces a large number of different substances, all of which are viscous liquids, containing a volatile solvent, which on evaporation leaves behind coherent, flexible, and usually transparent films. The most important classes are the oil-turpentine and the spirit varnishes. The varnish industry is an important and growing one, as shown by the census reports for 1900 and 1905 of the quantities and values of varnishes produced:

Quantity and value of varnish produced in 1900 and 1905.

Material.	1900.		1905.	
	Quantity.	Value.	Quantity.	Value.
	Gallons.	*Dollars.*	*Gallons.*	*Dollars.*
Oil and turpentine varnish	14,804,251	14,530,159	17,929,403	16,170,612
Alcohol varnish	553,432	921,169	1,542,562	2,203,645
Pyroxylin	171,127	187,626	458,361	562,629

It is not possible to determine from the census figures the quantity of raw materials used in varnish making. The above figures, however, give an idea of the relative quantities and values of the several kinds of varnishes made, and also show that the industry is steadily growing. In the manufacture of the alcohol varnishes the following quantities of alcohols were used in 1900 and 1905:

Quantity of alcohols used in varnish manufacture in 1900 and 1905.

Kind of alcohol.	1900.		1905.	
	Quantity.	Value.	Quantity.	Value.
	Gallons.	*Dollars.*	*Gallons.*	*Dollars.*
Grain alcohol	78,309	175,907	59,064	138,703
Wood alcohol	310,059	285,570	1,357,682	790,243

Thus you will see that before the denatured alcohol law became operative nearly all of the alcohol varnishes were made with wood alcohol. In the manufacture of collodion or photographic films and plastics the following quantities of alcohol were used in 1900 and 1905:

Alcohols used in the manufacture of collodion and plastics, 1900 and 1905.

Kind of alcohol.	1900.		1905.	
	Quantity.	Value.	Quantity.	Value.
	Gallons.	*Dollars.*	*Gallons.*	*Dollars.*
Grain alcohol...	7,015	16,837	8,557	20,858
Wood alcohol...	187,881	121,737	275,278	204,925

Here, too, wood alcohol has apparently been found to serve every purpose of the industry, and but little of the high-priced grain alcohol was used. From the preceding figures it is clear that varnish making affords a market for at least 2,000,000 gallons of alcohols per year, with the possibility of substituting a portion of the 7,000,000 gallons of turpentine, 13,000,000 gallons of petroleum oils, and smaller quantities of amyl alcohol or amyl acetate, which are now also used in making varnishes, much of them in spirit varnishes.

The manufacture of spirit varnish is in principle exceedingly simple, consisting of dissolving one or more resins in one or more volatile solvents. To hasten solution the resins are powdered and sometimes mixed with clean sand or powdered glass, and then are treated with alcohol, being stirred by hand or suitable machinery. The gums may be dissolved cold or by heat. The liquid at this stage is usually cloudy, as well as colored. To make the varnishes clear and brilliant they are allowed to settle for a long time and are then racked off, or preferably are filtered through a linen or muslin-lined wire basket fitted in the top of the varnish reservoir, taking suitable precautions to prevent the loss of solvent through evaporation. If a light-colored varnish is desired, it is made by filtering the varnish through animal charcoal in the form of coarse sand. If, on the other hand, it is desired to give the varnish a particular color, or in other words to make a lacquer, this color, which may be one of the anilin or vegetable dyes, is dissolved in the solvent and added to the finished varnish, or if a colored mineral is used which is insoluble in the varnish, the mineral as an impalpable powder is ground with a part of the varnish in a paint mill and then thinned with the remainder of the varnish.

Regarding the suitability of varnishes for special purposes, Livache, having regard only to the predominant resin, classifies spirit varnish as follows:

Gum.	Varnish used for —
Amber..............	Metals, photography, bookbinding.
Copal (fused).......	Bookbinding.
Copal (soft)........	Interior decoration.
Shellac..............	Articles subjected to wear and tear, trays, leather, cardboard, blackboards, French polish.
Shellac (blended)...	Colorless varnish.
Mastic...............	Toys, turned articles, pictures, cardboard, golden lacquer.
Sandarac...........	Pounce cabinet work, water colors, visiting cards, varnish to be used to thin down colors, golden lacquer, white spirit varnish, wood carving, metal plate work, photographic negatives.
Celluloid...........	Flexible articles, electrical insulation, photographic negatives, waterproof varnishes.

As a rule shellac is the basis of most of the resin-spirit varnishes, and to this is added mastic, sandarac, and other resins to give special properties. The alcohol used is usually about 90 per cent, though as weak as 80 per cent may be used, and for some varnishes 98 per cent alcohol is employed. The following formulæ are typical of various resin-alcohol varnishes:

Pale shellac polish:
    Orange shellac..pounds.. 90
    Alcohol...gallons.. 45
White shellac polish:
    Bleached shellac..pounds.. 60
    Alcohol...gallons.. 27
Copal varnish:
    Manila copal..pounds.. 160
    Venice turpentine...do.... 40
    Alcohol...gallons.. 36
Sandarac varnish (paper varnish):
    Sandarac..pounds.. 40
    Venice turpentine...do.... 30
    Alcohol...gallons.. 18
Mastic varnish for water colors:
    Mastic..parts.. 5
    Venice turpentine...do.... 2
    Alcohol..do.... 14
Mixed varnishes:
    (1) Manila copal..pounds.. 50
        Rosin ....do.... 30
        Burgundy pitch ....do.... 30
        Alcohol ....gallons.. 20

    (2) Copal...parts.. 60
        Alcohol ....do.... 6
        Ether ....do.... 10
        Turpentine ....do.... 40
Hat-stiffening varnish:
    (1) Orange shellac..pounds.. 7
        Sandarac ....do.... 2
        Mastic ....ounces.. 4
        Amber rosin ....pound.. ½
        Copal spirit varnish ....pint.. 1
        Alcohol ....gallon.. 1

    (2) Sandarac..pounds.. 30
        Rosin ....do.... 20
        Elemi ....do.... 10
        Alcohol ....gallons.. 12

These formulæ are sufficient to indicate the wide range of possible combinations and the large number of uses to which spirit varnishes are put.

Another class of products is known as pyroxylin varnishes. These are made by dissolving nitrated cotton or cellulose in alcohol—ether, amyl alcohol, or amyl acetate. Such varnishes are extensively used as fillers on wood, and for covering metals, leather, and other manufactured articles, as well as for the manufacture of photographic films. Their body may be improved for some purposes by mixing with resin varnishes. One of the most important uses of pyroxylin varnish is in the

preparation of collodion for photographic dry plates and films. For these films the following formula has been found satisfactory:

	Parts.
Methyl alcohol (97 per cent)	6. 5
Amyl alcohol	2. 5
Amyl acetate	1. 0

To 1 gallon of this mixture add 1 pound of pyroxylin. The films are made by pouring on glass plates and drying in a dry atmosphere.

The term "lacquers" is synonymous with spirit varnishes, though it is common to designate varnishes which are colored with dyes as lacquers. When varnishes or lacquers are ground with pigments, the product is known as enamel.

Methyl or wood alcohol has been by far the most generally employed alcohol in making spirit varnishes, and, therefore, grain alcohol which has been denatured with the general denaturants, methyl alcohol and benzin, or methyl alcohol and pyridin bases, will undoubtedly serve all practical purposes in the vast majority of cases. Indeed, such alcohol is undoubtedly to be preferred to methyl alcohol because, containing less methyl alcohol and other impurities, it is less injurious to health and less likely to be damaging to the articles upon which it is employed.

When we bear in mind, however, the varying nature of the materials from which varnish and lacquers are made, and think of the list of all kinds of materials, including wood, metals, paper, cloth, and finally photographic films, to which they are applied, which must not be affected by but rather preserved from change and injury by such varnishes, it is clear that these denaturants may not be in all cases the most suitable from either the technical or financial point of view. Hence to permit greater freedom of use to meet such conditions, the Commissioner of Internal Revenue has permitted a number of other denaturants which may be used in varnish making. Thus for lacquers, pastes, and varnishes from soluble cotton, 5 gallons of approved wood alcohol per 100 gallons of grain alcohol, or 2 gallons of approved wood alcohol and 2 gallons of benzol per 100 of grain alcohol, may be used. Five gallons of approved wood alcohol per 100 of grain alcohol may be used also for filaments of incandescent electric lamps, for watches and jewelry, for moldings and picture frames, for photographic dry plates, for shellac varnishes, and for shoe polish. For photographic collodion 100 pounds of sulphuric ether and 10 pounds of cadmium chlorid, or 3 gallons of 1.83 sulphuric acid and 1 gallon of 0.8 specific gravity kerosene, may be used. Should the varnish industry require it, no doubt the list of special denaturants will be extended.

From the foregoing brief statement it is seen that the present demand for denatured alcohol for use in the varnish industry is not great. Undoubtedly its use for this purpose will gradually increase, particularly if the production of turpentine decreases and alcohol can be sold at a price that will compete with light mineral oils.

USE OF ALCOHOL FOR POWER.

By R. M. STRONG, *United States Geological Survey.*

In discussing the use of alcohol for power, probably the clearest understanding of the subject can be obtained by comparing the action of gasoline and alcohol engines, as they are built on exactly the same principle, and supplied with their fuel in relatively the same manner.

Considering the heating values of gasoline and alcohol alone, it is obvious that the ratio of the consumption of alcohol to the consumption of gasoline per horsepower hour will be inversely proportional to their heating values, if used with an equal thermal efficiency. The low heating value of denatured alcohol, which corresponds

very closely to 94 per cent by volume ethyl alcohol, will average about 10,500 B. t. u. per pound, while the low heating value of 0.71 to 0.73 specific gravity gasoline will average about 19,100 B. t. u. per pound, making the above-mentioned ratio 1.8 to 1 by weight. But when alcohol and gasoline are used separately in a gasoline engine without change of compression and the engine is operated under the most advantageous conditions in each case, the ratio of alcohol consumption to gasoline consumption per horsepower is 1.6 to 1 by weight, while by raising the compression from that best for gasoline (about 70 pounds per square inch above atmosphere) to that best for alcohol (about 180 pounds per square inch above atmosphere) the ratio by weight is further reduced to 1.2 to 1. The corresponding ratio by volume is 1 to 1, owing to a difference in the specific gravity of the two liquids, and the thermal efficiency ratio is 1.5 to 1.

By using alcohol then in an alcohol engine, with high compression (about 180 pounds per square inch above atmosphere, much higher than can be used for gasoline on account of preignition from the high temperatures produced by compression) the consumption of alcohol, in gallons per horsepower per hour, can be made to be practically the same as that for gasoline in a gasoline engine operating under the same conditions of best load and other affecting conditions. These variables, which are many and complex, are such that it is possible to use from one-half again to three times as much fuel as is necessary for any given load. When an engine is running at maximum load and governing for constant speed, only about 50 per cent of excess alcohol can be supplied without causing the engine to slow down and stop, while at half load about two and a half times the amount needed can be supplied before the engine will show any outward change in operation, except, possibly, that an odor of alcohol or formalin in the exhaust will indicate to the operator that an excess of fuel is being supplied. An oversupply of alcohol does not produce an exhaust of black smoke as does an excessive oversupply of gasoline, yet the per cent excess of alcohol and gasoline that can be used and not be detected from outside appearances is approximately the same. The maximum per cent excess of gasoline that can be used at maximum loads is a little greater than that for alcohol, while for light loads a greater excess of alcohol is likely to be used, because of the absence of smoke, which with gasoline would indicate to the operator that fuel was being supplied far in excess of that needed.

With the stationary engines which govern for constant speed, the following general rule of adjustment for minimum fuel consumption will hold in almost every case for loads from about one-half to the maximum load. Adjust the time of ignition and fuel needle valve so that a minimum possible amount of fuel is admitted per stroke without reducing the speed of the engine when the maximum load it is called on to carry is applied. This will give the minimum fuel consumption for that particular load and in most cases the fuel needle valve and igniter settings will be about the best that can be determined for all other loads down to about a half load. In order to obtain the minimum fuel consumption for the gradation of loads from maximum down to about half load, a continued reduction of fuel supply with best time of ignition will be necessary— that is, the smallest quantity of fuel that can be supplied per stroke and carry the load will give the minimum fuel consumption when the best time of ignition, which changes with the quality of the mixture, is used. With the hit-or-miss method of governing, even though the number of fuel admissions per minute be a maximum (that is, the number of cut-outs are only sufficient to insure certainty of operation) and though nearly every first fuel admission after cut-outs misfires from being too weak a mixture, the above adjustment will be the most economical.

With the throttling method of governing, where the disturbing effect of the governor is eliminated, the above rule will hold for all loads down to about one-quarter load, and lower in some cases.

The consumption of 1 pint of gasoline per brake horsepower per hour may be given as a fair average figure for an engine when operated at about rated load and under

reasonably favorable conditions, both from the standpoint of the skill of the operator and the mechanical condition of the engine. A like figure of 1 pint per brake horse-power per hour can reasonably be expected for alcohol when a like understanding of its use is obtained and engines as well adapted to the economical use of alcohol are constructed. Special tests show that it is possible to obtain a like fuel consumption figure of 0.8 of a pint per brake horsepower per hour for each fuel, when the most favorable practical conditions are obtained for the use of alcohol and for the use of gasoline in stationary engines of from 10 to 15 horsepower running at speeds of from 250 to 300 revolutions per minute.

The speed and size of the engine—that is, the size of the engine cylinder—has a certain influence on the fuel economy and thermal efficiency. A large cylinder engine can be made to be more efficient than a small one, probably because of the effect on the heat loss of the decrease in ratio of cooling surface to volume of charge or heat liberated. But the difference from this cause, which has been fairly well determined by experiment, is comparatively small when compared with the effect of the difference in mechanical conditions, such as leakage and friction, which are likely to be much greater in proportion for a small engine than a large one. For similar reasons it is also harder to maintain compression with a small engine than a large one, which difficulty increases with the degree of compression. Hence a small alcohol engine is likely to be less satisfactory than a small gasoline engine, depending entirely, however, on the perfection of construction and the skill of the operator.

The question of carburation is a rather complex one, though the principle of the ordinary carburetor is that of the simple atomizer, and any effective atomizing device will make a good carburetor if the other influencing conditions are properly controlled. Alcohol is less volatile than gasoline, and hence harder to vaporize. Under similar conditions more alcohol will pass into the cylinder of the engine in the form of a spray, but both may be completely vaporized by the heat from the cylinder walls and the mixture of hot gases left in the clearance space from the preceding explosion, or by the heat developed during compression. However, a greater excess of fuel and higher velocity of air through the carburetor is required to obtain an explosive mixture on starting an engine with alcohol than with gasoline. This requires a higher rotative speed, which may also tend to assist the vaporization of the spray carried by the air, by giving less time for the dissipation of the heat of compression to the cylinder walls. Throttling the charge, which reduces the pressure at which vaporization takes place, also assists the process, so that with a combination of these conditions it is not difficult to start a small alcohol engine "cold." To start a large alcohol engine, however, which can not readily be revolved rapidly by hand, will require some mechanical means to bring it up to speed, or this may be accomplished by priming with gasoline. Hot passageways and cylinder walls assist in vaporizing the fuel at the start, at least, but are not necessary if the previously mentioned conditions are maintained.

Most gasoline engines with good carburetors run equally well with alcohol and with gasoline from the standpoint of operation, with the exception only of a difference in ease of starting and certainty of operation under conditions of variable speed. The only alteration required for the use of alcohol in a gasoline carburetor, if any, will be in the size of the fuel passageways. The fuel needle valve must be capable of being opened twice as wide for alcohol as for gasoline and the spray nozzle must not be restricted to just the size that is required to supply the proper quantity of gasoline. The fuel passageways in a carburetor can usually be easily drilled out, and so far as producing power at constant speed is concerned, the engine will be just as serviceable with either fuel. This change need not be such as to affect the consumption of gasoline, but with this change alone the consumption of alcohol will be from one and a half to twice as much as the consumption of gasoline for the same power.

In some cases a gasoline engine may be altered further, so that a sufficiently high compression is secured to make it possible to reduce the consumption of alcohol in gallons per horsepower per hour to an equality with that for gasoline before the engine was changed. This plan precludes the further use of gasoline, as it can not be used satisfactorily with compressions much in excess of from 70 to 75 pounds per square inch above atmosphere.

The compression may be most easily changed by lengthening the connecting rod, which changes the position of the piston at the extreme end of the compression stroke. This can only be done, however, when the engine is not counterbored and the shape of the clearance space is such that the piston will not strike the cylinder head or valves. The type of connecting rod usually used for stationary engines can be lengthened very easily by putting liners between the crank pin end of the connecting rod and the crank brasses, but if the cylinder is counterbored or the piston strikes the head of the cylinder or the valves, a new piston or cylinder head with smaller clearance space should be made. Attaching plates to the piston or cylinder head is seldom, if ever, satisfactory. The arrangement of the valve-actuating mechanism often determines the position of the valves, which may be such that a small enough clearance space can not be secured without almost an entire redesign of the engine. Furthermore, with the increased compression required for the economical use of alcohol, the maximum pressure from explosions or combustion will increase and will be as high as 600 or 700 pounds per square inch for maximum load when the compression is raised to 180 to 200 pounds per square inch above atmosphere. Stationary gasoline engines, especially of the horizontal type, are not usually built heavy enough to withstand any such pressures continuously, so that while it may be quite possible to raise the compression of a gasoline engine sufficiently to convert it into an economical alcohol engine, it is not always advisable to do so. This is especially true if the difference in cost of fuel is a comparatively small consideration, as is quite often the case.

When alcohol is used in a gasoline engine with the best gasoline compression, the maximum power of the engine is increased about 10 per cent, while the maximum power of an alcohol engine of the same cylinder size, stroke, and speed will be about 30 per cent greater with the best alcohol compression. Owing to the higher explosion pressures, however, an alcohol engine should be built heavier than a gasoline engine and the weight per horsepower may be less.

A question that is asked by almost every operator is, "What effect has the temperature of the jacket or cooling water on the fuel economy and thermal efficiency of an alcohol or gasoline engine and what is the best jacket temperature?" The only safe answer to make to this question is, "The best jacket temperature is that which gives the best results." Many tests go to show that the temperature of the cooling water has but little, if any, appreciable effect on the fuel economy and thermal efficiency of the engine by controlling to a greater or less extent the temperature of the cylinder walls. Jacket temperature may, however, have a great effect on the performance of the engine from its effect on other conditions than cylinder wall temperature. These conditions, in all probability, can be reached in an entirely different way and are not necessarily dependent upon the jacket temperature. It has been pretty well established that when jacket temperature affects the fuel consumption of an alcohol or gasoline engine, it is more from effect on cylinder lubrication, fit of piston and valves, and carburation than from any direct effect of heat loss to the cylinder walls. A hot jacket may stop leakage by the piston and valves by making them tight from expansion, or it may make a tight piston stick or bind, and warp valves or valve seats that are tight when the cylinder walls are kept cold. If the cylinder oil used is thick and heavy, it will flow better when the cylinder is kept hot, but thin oil is more likely to run out and leave the piston dry. If the design of

the carburetor is such that efficient carburation and uniform mixtures depend on hot passageways, a "hot jacket" will help counteract the effect of poor or limited design and the ultimate effect of a "hot jacket" on the fuel consumption is, for this reason, likely to be greater for alcohol than for gasoline.

The temperature to which the jacket water can be raised is sometimes limited by the design of the engine being such that for a high temperature of jacket water the cooling system is not effective in keeping all the parts below the temperature that will cause preignition. It is not at all necessary to keep the temperature of the jacket water high, if the design and construction of the engine is right to begin with and suitable cylinder oil is used. A "hot jacket" may, however, be a good remedy to use for many ills that may occur from poor or limited design and construction.

Similarly, preheated air may be of great benefit when the carburation is ineffective or the mixture is not uniform in quality, but it is not necessary to preheat the air to obtain the best results with alcohol, and if heated to any great degree it will diminish the capacity of the engine to a detrimental extent.

The increased difficulty of starting an engine with alcohol and the increased uncertainty of operation under variable speed conditions has suggested the use of gasoline and alcohol in a double carburetor, which mixes the vapors of sprays of the two fuels with air in the usual manner. With such mixtures the thermal efficiency, however, varies from that for alcohol alone (maximum 40 per cent) with best compression, to that for gasoline alone (maximum 28 per cent). These thermal efficiencies are calculated from the indicated horsepower and low heating value of the fuel. The total fuel consumption is never less than the like figure for either fuel alone (minimum =0.8 pint per brake horsepower per hour). The difficulties of starting the engine decrease with the increase in gasoline used. There is no advantage in using mixtures of gasoline and alcohol except in starting and certainty of operation under conditions of variable speed, the advantages of which should, if possible, be obtainable through some other means, such as better design of engine and carburetor. Further, the use of gasoline in any quantity does away with many of the advantages that are obtained from the use of alcohol alone, such as safety and absence of disagreeable odors.

So far the alcohol referred to in this discussion has been the completely denatured alcohol, which by law must be greater than 90 per cent ethyl alcohol by volume. We are not, however, limited to the use of 90 per cent alcohol from the engine standpoint. Even 50 per cent alcohol can be used, though not very satisfactorily. An engine is very hard to start with 50 per cent alcohol and is very uncertain in its operation; that is, the operating conditions are very limited and the thermal efficiency decreases rapidly with increased dilution. The rate of decrease of the thermal efficiency with decrease in per cent alcohol, however, is not constant, but increases more and more rapidly as the alcohol becomes more dilute. From 94 per cent to 80 per cent alcohol, however, the consumption of pure alcohol is about the same and the total consumption is very nearly directly proportional to the increase in per cent of water. The significance of this is that if 80 per cent alcohol can be manufactured for 15 per cent less per gallon than 90 per cent alcohol, it becomes of commercial advantage to use the former grade, provided the difference in cost of handling the greater bulk does not offset the gain in cost of manufacture. It is possible even that the use of 70 per cent alcohol or lower grades will prove to be cheaper, but the use of these more dilute alcohols is limited to a certain extent by the difficulty experienced in starting an engine using them and in the increased uncertainty of operation, especially under conditions of variable speed.

With the development of the processes of manufacturing alcohol, we may expect a notable advance in the design of alcohol engines and carburetors. The effect of these developments may bring the general use of alcohol well within the scope of

commercial requirements for use in stationary engines, at least, where the difficulty of starting is not serious and the certainty of operation is favored by fairly constant speed and load conditions. The economical use of alcohol for variable speed engines operating under widely varying conditions, such as the automobile engine, is not at all improbable. In fact, alcohol motor trucks and taxicabs are being tried out and with considerable success. Alcohol locomotives for mine haulage and other similar work are also being built.

From the standpoint of fire risk alcohol has a decided advantage over gasoline, or possibly kerosene. The relative fire risk from the use in motors and from the handling and storage of gasoline, alcohol, and kerosene has not yet been definitely established and the restrictions at present placed on alcohol may be unfair and hinder its adoption as a fuel to a considerable extent.

The development of kerosene engines and those that will use the cheaper distillates and crude oils, the latter not costing over 4 cents per gallon in many districts, may affect the general use of alcohol for some time to come, but as yet too little is known of the extent to which these cheaper oils may be adapted to engine use or engines adapted to use them economically, to predict the extent to which alcohol may compete with them.

Referring to the bulletins on the use of denatured alcohol in internal-combustion engines, which were published by the Office of Experiment Stations of the United States Department of Agriculture, it is a pleasure to state that such of the work at the fuel-testing plant of the United States Geological Survey as necessarily supplemented work already done in order to obtain and define the conditions necessary for the most economical use of alcohol in special alcohol engines corroborates the figures obtained by Professor Lucke and Mr. Woodward and fulfills many of the predictions made by them from their work on gasoline engines adapted to a certain extent only to the use of alcohol, which equipment was the best available in this country at the time. This work is of great value in itself from the point of view from which it was made and has been of considerable assistance in later research, which, however, may throw a little different light on the subject, causing an apparent disagreement of results that does not really exist.

LEGISLATION AND REGULATIONS.

LEGISLATION PROVIDING FOR THE USE OF INDUSTRIAL ALCOHOL.

By John G. Capers, *Commissioner of Internal Revenue.*

The tax on alcohol in the shape of distilled spirits alone in the United States produced during the past fiscal year in round numbers $140,000,000 of revenue, or nearly 60 per cent of the total sum derived from internal taxes and more than 20 per cent of the entire receipts of the Government from all sources. No other single substance or article bears anything like the same burden of tax either in customs or internal taxation at the present time. We of the present generation are accustomed to look upon the spirit tax as a perfectly legitimate and highly essential source of revenue, yet there are many persons living to-day who can readily revert in their recollection to a time when no tax whatever was laid upon alcohol in any form, and distilled spirits were produced from any source whatsoever without government supervision, and sold for any purpose as freely as any other article of merchandise. In fact, for more than half of its existence as a nation the Republic derived no revenue from this source, which is now considered so important that its possible future curtailment by the prohibition movement, which is sweeping over the country, is already becoming a matter of grave concern to the lawmakers.

In the earlier days it was considered as an emergency tax, only to be resorted to for temporary exigencies, such as a state of war. At the close of the civil war, however, in 1868, a tax of 50 cents a gallon was retained; this was raised to 70 cents in 1872, again to 90 cents in 1875, and to $1.10, the present figure, in 1894. This is a rate that is higher than any other of the leading nations imposes, with the single exception of Great Britain, where the tax is nearly twice as high as our own rate.

While the rate has varied all the way from 20 cents up to $2 per proof gallon in the various laws, in all other respects there has been but little difference, the tax being levied upon distilled spirits, from whatever source they may have been produced and whatever the purpose for which the spirits were used or intended.

Whenever privileges have been granted, therefore, in regard to the tax-free use of alcohol, it has been in the form of laws allowing exemptions from taxation. Naturally all of these laws are subsequent to 1862, since prior to that time, with the exception of the two brief periods, 1791–1800 and 1813–1818, there was no tax from which to grant exemption. Mr. David A. Wells, the writer on economics, estimated that in 1860 about one-third of the total consumption of distilled spirits was used for industrial purposes. The effect of the heavy taxes imposed at the beginning of the civil war on the industries using alcohol is graphically set forth by Wells,[a] as follows:

"The immediate effect of this imposition and rapid increase of internal taxes on distilled spirits was a series of industrial and commercial phenomena, more remarkable than anything of the kind before recorded in economic history. In short, the influence of these taxes was to entirely and rapidly revolutionize great branches of domestic industry, and in some instances to utterly destroy them."

I come now to a consideration of the various forms of relief that have been granted by Congress from time to time, all of which, as already stated, have taken the shape of exemptions from tax of alcohol or distilled spirits when used for certain specified purposes.

The law of June 7, 1906, generally known as the Denatured Alcohol Law, which went into effect January 1, 1907, was the first successful legislation in the direction of a general use of alcohol free of tax in the arts and industries in the United States. Prior to that certain limited legislative permits had been granted for the free use of alcohol for special purposes. The first of these, in point of time of enactment, is embodied in section 3297, Revised Statutes, as follows:

"Sec. 3297. The Secretary of the Treasury is authorized to grant permits to any incorporated or chartered scientific institution or college of learning to withdraw alcohol in specified quantities from bond without payment of the internal revenue tax on the same, or on the spirits from which the alcohol has been distilled, for the sole purpose of preserving specimens of anatomy, physiology, or natural history belonging to such institution, or for use in its chemical laboratory: *Provided*, That application for permits shall be made by the president or curator of such institution, who shall file a bond for double the amount of the tax on the alcohol to be withdrawn, with two good and sufficient sureties, to be approved by the Commissioner of Internal Revenue, and conditioned that the whole quantity of alcohol so withdrawn from bond shall be used for the purposes above specified, and for no other, and that the said president or curator shall comply with such other requirements and regulations as the Secretary of the Treasury may prescribe. And if any alcohol so obtained is used by any officer, as aforesaid, of such institution for any purposes other than that above specified, then the said officer or sureties shall pay the tax on the whole amount of alcohol withdrawn from bond, together with a like amount as a penalty in addition thereto."

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[a] Report No. 7097, Ways and Means Committee, Fifty-ninth Congress, on H. R. 24816.

The privileges of this section were extended by the act of May 3, 1878, as follows:

"Sec. 3297a. That the Secretary of the Treasury be, and is hereby, authorized to grant permits, as provided for in section thirty-two hundred and ninety-seven of the Revised Statutes of the United States, passed at the first session of the Forty-third Congress, to any scientific university, or college of learning created and constituted such by any State or Territory under its laws, though not incorporated or chartered, upon the same terms and subject to the same restrictions and penalties, already provided by said section thirty-two hundred and ninety-seven: *Provided further*, That the bond required thereby may be executed by any officer of such university or college, or by any other person for it and on its behalf, with two good and sufficient sureties, upon like conditions, and to be approved as by said section is provided."

These laws are in full force and effect at the present time, not having been affected or curtailed in any way by the Denatured Alcohol Law. The regulations under which they operate are contained in a six-page circular, known as Internal Revenue Circular No. 715, copies of which may be had on application.

Section 3464 of the Revised Statutes provides as follows:

"Sec. 3464. The privilege of purchasing supplies of goods imported from foreign countries for the use of the United States, duty free, which now does or hereafter shall exist by provision of law, shall be extended, under such regulations as the Secretary of the Treasury may prescribe, to all articles of domestic production which are subject to tax by the provisions of this title."

Under this section, distilled spirits, including alcohol, are withdrawn from bonded warehouse free of tax for the use of the United States.

Of the various purposes for which alcohol is withdrawn, the greatest quantity is used in the manufacture of ammunition for the army and navy. The various chemical laboratories in different branches of the government service obtain alcohol free of tax under its provisions, which are contained in Department Circular No 104.

In the annual report of the Commissioner of Internal Revenue the quantities of alcohol and distilled spirits withdrawn free of tax under sections 3297 and 3464 are grouped together. For the fiscal year ended June 30, 1908, these quantities were as follows:

	Taxable gallons.
Bourbon whisky	1,609
Rye whisky	577
Alcohol	1,352,507
High-proof, neutral, or cologne spirits	241,325
Total	1,596,019

Another law which has been in operation since the passage of the act of March 1, 1879, provides for the use of alcohol (not as alcohol per se, but in the form of a distillate from a fermented mash) in the manufacture of vinegar. The law is embodied in Revised Statutes, 3282, extended by the act of March 1, 1879, and the regulations for its execution may be found in Regulations No. 7, pages 214–217. No accounting is required of vinegar manufacturers operating under this law, so the office has no basis for determining with any accuracy the quantity of alcohol used for the purpose. No change was made in this law by the passage of the Denatured Alcohol Law, except that vinegar could also be made from alcohol, free of tax, under the latter, provided a suitable denaturant could be found. No information has been received by the internal-revenue office, however, to indicate that vinegar manufacturers in general were desirous of operating under the new law. In Germany the manufacture of vinegar absorbs more denatured alcohol than any other single line of manufacture.

The law generally known as the McKinley tariff act, passed in 1890, provided for the withdrawal of grape brandy, free of tax, for use in the fortification of pure sweet wines. The amended laws and regulations are contained in Regulations No. 28.

This law is in full force and effect and there were withdrawn from bond under its provisions, free of tax, in the fiscal year ended June 30, 1908, 4,380,017 taxable gallons of grape brandy.

In the annual appropriation bill for the Department of Agriculture in 1891 there is a clause providing for the withdrawal of alcohol of a strength of not less than 160° proof for use solely in the manufacture of sugar from sorghum. This law still stands upon the statutes, but no advantage has ever been taken of its privileges.

To make the story of tax-free alcohol complete, there remains only for mention the withdrawal of distilled spirits, free of tax, first, for exportation, and, second, for transfer to manufacturing warehouses, bonded for use in the manufacture of articles for exportation, both of which have been in operation for a long time under quite a complex system of control, provided for in Regulations No. 29. For the fiscal year ended June 30, 1908, the quantity of distilled spirits of all kinds withdrawn from bond, free of tax, was as follows:

Taxable gallons.

For exportation as distilled spirits	1,383,151
For the manufacture of articles for exportation	157,307

I have said that the present Denatured Alcohol Law was the first successful legislation in the direction of a general use of alcohol free of tax in the arts and manufactures. I said successful advisedly, for I now come to an unsuccessful attempt of this kind, which eventually acted rather as a hindrance than as a help to the movement. I refer to the legislation enacted as a part of the tariff law of August 28, 1894, known as the "Wilson bill," section 61 of which provided as follows:

"SEC. 61. Any manufacturer finding it necessary to use alcohol in the arts, or in any medicinal or other like compound, may use the same under regulations to be prescribed by the Secretary of the Treasury, and on satisfying the collector of internal revenue for the district wherein he resides or carries on business that he has complied with such regulations and has used such alcohol therein, and exhibiting and delivering up the stamps which show that a tax has been paid thereon, shall be entitled to receive from the Treasury of the United States a rebate or repayment of the tax so paid."

It will be seen that the provisions of section 61 were general enough, covering a much more extensive use of alcohol than is permitted in the present law. In fact it was entirely too "general" in terms and scope, and this led to its undoing, as the then Commissioner of Internal Revenue, Mr. Miller, with the approval of the Secretary of the Treasury, Mr. Carlisle, refused to carry it into effect on the ground that the language was so indefinite that it would be impossible to execute it with safety to the revenues of the Government. The manufacturers who were the beneficiaries of the law then carried the matter into the courts, by using the alcohol, keeping the necessary accounts just as if regulations had been issued, and then making formal application for a refund of the tax. This was refused, and they then brought suit to collect the moneys involved. These claims for refund, speaking at large, probably aggregated a sum of about $10,000,000.

The legal battle for this sum was fought out by means of a test case known as the "Dunlap hat case," which was first brought in the Court of Claims, where the claimant lost, then tried in the Supreme Court, where it was decided favorably to the Government by a divided decision of the court, thus putting an end to the execution of this law, which really retarded the progress of the movement for over ten years. During the same time that the case was passing through the courts Congress also took action, repealing section 61, but providing, in the same act, for a joint select committee to consist of three Senators and three Representatives, who were directed to make a study of the entire subject of the use of alcohol free of tax, and report their conclusions to Congress.

This committee held meetings and took testimony in Washington and in most of the large cities in the United States, and made its recommendations to Congress, the report consisting of a volume of nearly a thousand pages of printed matter.[a]   Their recommendations were not unanimous, two members expressing the opinion that the use of alcohol tax-free in the arts and industries should be provided for by Congress just as soon as the condition of the public revenues would allow, while at the same time they indicated that in their judgment that time had not then arrived, and this view was concurred in by a majority of the committee.   One member made a separate report, in which he expressed himself as opposed to either present or future legislation of this character.

The attitude of the officials of the Treasury Department toward legislation of this kind, although changing somewhat from time to time, has been generally unfriendly. This was natural for obvious reasons.   It is easy to understand that officers specifically charged with the duty of collecting the revenues and preventing frauds against them would be apt to look with more or less disfavor upon legislation which would necessarily result in increasing the opportunities for fraud against the revenues derived from one of the most important sources.   It does not appear, however, that at any time in the period covering the agitation of the question was any disposition shown to shirk the responsibility of executing a measure for the free use of alcohol in the arts and manufactures provided it carried sufficient authority for its execution without endangering the collection of the revenues.

A list is appended of the various measures submitted to the internal-revenue office by Congress, and which were returned to the latter body with criticisms and comments concerning them from an administrative standpoint (see p. 151).   It may be stated, also, that the unsuccessful legislation contained in the tariff bill of 1894 was not submitted to the Treasury prior to its enactment.   The liberal disposition of the Department toward the movement in later days is shown by the fact that a draft of a bill was submitted to the Ways and Means Committee of the House at the session prior to the enactment of the present law, with the assurance that the Treasury Department felt assured that the legislation, if enacted in the form of the bill submitted, or along similar lines, could be executed without serious risk of imperiling the collection of the revenues, or unduly enhancing the cost of said collection.   Aside from the question of possible fraud, however, Congress was at that time facing the prospect of a serious deficit in the receipts as a whole, and it was not until the convening of the first session of the Fifty-ninth Congress that the matter received any earnest consideration by the committee.

The history of the passage of the present law is so recent that it is probably well known to most of you, and I need not dwell upon it.   The hearings of parties interested in its provisions, and the active campaign organized in its behalf by a committee of the manufacturers using alcohol in various ways; the placation of temperance organizations, which were at first inclined to bring their powerful batteries of opposition to bear upon it, but which were persuaded that it was actually a temperance measure, so that their opposition was changed to advocacy; the enrollment of the Farmers' Grange among its supporters on the plea that it would result in great benefit to the agriculturists, both directly and indirectly; its mention by the President in one of his special messages as a possible check to the commercial rapacity of the petroleum octopus, were all chapters in the story of its rapid progress to successful enactment.

The law, as finally enacted, imposes a much greater degree of authority and responsibility upon the executive officers than was recommended or desired by the then Commissioner of Internal Revenue.   His position was stated before the House committee in the hearings held upon the bill.   He did not desire the scope of power and authority given him under the bill as it passed the House, and he much preferred a bill he had

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[a] Senate Report No. 411, Fifty-fifth Congress.

prepared, which was given in the hearings before the Senate Finance Committee. This bill, which was shaped more on the lines of existing laws concerning internal-revenue taxes, read into the law itself many provisions which, in the law as passed, were left to the discretion of the administrative officers. The House bill was enacted, however, practically unchanged, Congress evidently believing that its operation should be mainly governed by regulation, as being more elastic and adaptable to changing commercial conditions than if the provisions were more rigidly fixed by legal enactment. This tendency is still further manifest in the amendatory act of March 2, 1907, which extends the scope of the law without in the least curtailing the administrative authority. The Commissioner of Internal Revenue, with the approval of the Secretary of the Treasury, determines absolutely the character of the denaturing materials and the system under which the denatured alcohol is handled and sold. The authority vested in the Treasury officers carries with it an equal measure of responsibility; and whether it was the intention of Congress or not, it has certainly brought about this result, that these officers have taken, ever since its enactment, a most intimate and anxious interest in its success. Recognizing the fact that the principal countries of Europe have allowed the tax-free use of alcohol for industrial purposes for many years, and that their experience could not fail to prove of value, an investigation of the various systems in operation there was made during the summer of 1906 by Mr. Yerkes and Hon. E. J. Hill, member of the Ways and Means Committee of the House, who had taken a very active part in the passage of the law, and who had urged legislation of this kind for many years in behalf of the large manufacturing interests of the New England States. The information obtained was of great value in the preparation of the regulations and the inauguration of the law, which went into operation in January, 1907.

The amendatory act took effect September 1, 1907, and bearing in mind the evident intent of Congress to extend the benefits of the legislation, which had already proven of great value to manufacturers, to the agricultural community by promoting the production of alcohol directly from crops and by-products of crops by the farmer himself, it was endeavored to make this practicable by relieving the new class of denatured-alcohol distilleries thus provided for of most of the restrictions placed upon the larger distilleries. At the same time the regulations concerning the handling and sale of denatured alcohol were modified in many ways, going as far in the direction of a liberalization of these regulations as the experience of the office in its six months' execution of the law seemed to justify.

It has been a matter of much disappointment that there has been no disposition shown so far by the farmers to avail themselves of the benefits extended to them by the law and regulations, but it is hoped that this work of education and experiment in their behalf by the Department of Agriculture will soon bear fruit.

Finally, an extended use of denatured alcohol in our country may be brought about by the legislatures of the various prohibition States passing laws to authorize the reopening of the idle, and in many instances expensive, distilleries, under such restrictions as would provide that their operation should be under State as well as National official inspection and surveillance for the purpose of turning practically all of the product into denatured alcohol for manufacturing and industrial purposes.

BILLS INTRODUCED IN CONGRESS, PROVIDING FOR THE REMISSION OR REBATE OF TAX ON ALCOHOL USED IN THE INDUSTRIAL ARTS, ETC.

H. R. 5082 (47th Cong., 1st sess.), to provide for the use of free alcohol in the industrial arts. Debates (Cong. Rec., vol. 13, pp. 5325, 5360–5364).

H. R. 901 (49th Cong., 1st sess.), authorizing the use of methylated spirits free of tax.

H. R. 10415 (49th Cong., 1st sess.), providing for use of alcohol, free of tax, in the arts and manufactures.

H. R. 9051 (50th Cong., 2d sess.), providing for the use of alcohol, free of tax, in the industrial arts.

H. R. 7977 (53d Cong., 2d sess.), providing for the establishment of warehouses for the manufacture of articles with alcohol, free of tax.

H. R. 4864 (53d Cong., 2d sess.), proposed Senate amendments providing bonded storerooms for manufacture of certain articles with alcohol, free of tax.

H. R. 886 (54th Cong., 2d sess.), proposed Senate amendments providing for bonded premises on which articles may be manufactured with tax-paid spirits with benefit of drawback.

H. R. 13302 (56th Cong., 2d sess.), to encourage the exportation of manufactured articles in which alcohol is a constituent.

REGULATIONS RELATING TO INDUSTRIAL DISTILLERIES.

By Wales Hubbard, *Internal Revenue Service, Treasury Department.*

Section 4 of the supplemental act of March 2, 1907, relating to denatured alcohol, provides:

"That at distilleries producing alcohol from any substance whatever, for denaturation only, and having a daily spirit-producing capacity of not exceeding one hundred proof gallons, the use of cisterns or tanks of such size and construction as may be deemed expedient may be permitted in lieu of distillery bonded warehouses, and the production, storage, the manner and process of denaturing on the distillery premises the alcohol produced, and transportation of such alcohol, and the operations of such distilleries shall be upon the execution of such bonds and under such regulations as the Commissioner of Internal Revenue, with the approval of the Secretary of the Treasury, may prescribe, and such distilleries may by such regulations be exempted from such provisions of the existing laws relating to distilleries as may be deemed expedient by said officials."

Under the authority conferred by this section of the act, regulations[a] have been issued governing the operation of distilleries of this class, and exempting such distilleries from many of the provisions of existing law relating to the distillation of spirits· As distilleries of this class may produce alcohol "from any substance whatever," and in order that the regulations, and the exemptions made, may be fully understood, the general provisions relating to the registry, survey, and operation of distilleries, especially to those in which grain or molasses is used, will be first briefly mentioned.

Under our revenue laws, distilleries are divided into three general classes, namely, grain distilleries, molasses distilleries, and fruit distilleries.

The spirit-producing capacity of grain and molasses distilleries is determined from the kind of materials to be used, the mode of mashing and fermenting, and the capacity of the mash and fermenting tubs; 25 gallons of mash is held to represent not less than 1 bushel of grain, and in estimating the number of dry inches to be allowed for fermentation from 3 to 10 inches (according to the kind of grain used) may be allowed for tubs 5 feet or less in depth, and a proportional allowance for tubs of larger size.

Fruit distilleries (which, under authorized regulations, are exempt from certain requirements applicable to grain and molasses distilleries) are surveyed according to the kind and spirit-producing qualities of the material to be used and the number of boilings that may be made in twenty-four hours, as determined from the kind, size, and working capacity of the stills employed.

Ordinarily, the working capacity of a still is about 90 per cent of its cubic contents, about 10 per cent being allowed for ebullition. Where the spirits produced require doubling, about three-tenths of the working capacity of the still is allowed for this

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[a] Regulations No. 30, United States Internal Revenue.

purpose. The number of boilings that may be made in twenty-four hours depends somewhat on the kind of heat used, whether steam or furnace, and, if the latter, upon the size and heating surface of the still. Usually seven boilings can be had in twenty-four hours, and in some instances a greater number.

The fermenting period, as to grain and molasses distilleries, is limited by law to seventy-two hours for sweet-mash distilleries, ninety-six hours for sour-mash distilleries, and one hundred and forty-four hours for molasses distilleries producing rum.

Molasses distilleries producing alcohol or other high-proof spirits by continuous process are surveyed either as sweet-mash or as sour-mash distilleries, according to the method of fermentation adopted. No limitation as to the fermenting period is imposed in the case of fruit distilleries.

All surveys are based on the period of twenty-four hours. All grain is computed on the basis of 56 pounds to the bushel, and the quantity of beer, per bushel, is reckoned as 45 gallons for sweet-mash distilleries and 60 gallons for sour-mash distilleries. In the case of molasses distilleries, 7 gallons of beer are allowed for each gallon of molasses.

In surveying distilleries, however, no fixed rule is adopted for determining the yield of spirits per bushel, or gallon, as the yield is found to vary according to the quality of the material used and the kind of still employed. The yield, however, is found to be, as to grain, about 3.5 to 4 gallons per bushel at ordinary distilleries, and from 4 to 4.5 plus at better equipped houses.

The yield from molasses is found to be from 85 to 90 per cent, i. e., 1 gallon of molasses should produce not less than 0.85 gallon of proof spirits.

Proof spirit is held to be that alcoholic liquor which contains one-half of its volume of alcohol of a specific gravity of 0.7939 at 60° F.

Registry of stills, filing of notices, etc.—All stills, to be used for distilling spirits, must be registered with the collector of the district in which they are to be used, and the distiller, before commencing business, must file with the collector a notice, giving, among other things, the name and residence of every person interested or to be interested in the business; the kinds of stills, mash tubs, fermenters, and receiving cisterns to be used, and the capacity of each; the mode of mashing and fermenting; the kind of materials to be used, the quantity to be used each day, and the estimated number of proof gallons of spirits to be produced each day of twenty-four hours.

The notice must also fully describe the distillery premises and the purpose for which each of the buildings situated thereon is to be used. A modified form of notice, however, is prescribed in the case of fruit distillers. Except as to fruit distillers and the smaller class of grain distillers, a diagram of the premises must also be furnished, showing the location of the several buildings and the arrangement of all stills, tubs, pipes, etc.

The distiller, if other than a distiller of fruit, must be the owner in fee of the premises, or must file the written consent of the owner to the use of the premises for distilling purposes. He must provide a meal room, cistern room, and warehouse, to be in charge of the designated officer and to be kept securely locked by him during his absence.

All wooden stills and all doublers, tubs, and cisterns must be so constructed and arranged as to have a clear open space around them, so as to be easily accessible to the inspecting officer. Pipes for conveying beer, low wines, spirits, or water must each be painted a distinctive color, indicating the purpose for which used, and must be exposed to view. All openings of tubs, cisterns, and pipes must be provided with locks, as also the pipes used for conveying spirits to the cistern room.

Certain other requirements respecting the construction and arrangement of distilleries are imposed, and certain restrictions are placed on the business of distilling, respecting the location of the premises, and the time and manner of operating the distillery.

Distiller's bond—Assignment of storekeeper.—After the distillery has been surveyed and its capacity determined the distiller is also required to file with the collector a bond in the prescribed form with satisfactory sureties, and after having duly qualified he may operate his distillery, subject to the requirements and limitations imposed. A storekeeper or storekeeper-gauger must be assigned to each grain or molasses distillery, and no such distillery can be lawfully operated in his absence.

Materials used and spirits produced.—All grain or molasses received on the premises, and all such materials before being used must be carefully weighed or measured by the officer, and a careful record must be kept showing the exact time when each fermenting tub is filled or emptied. In the case of fruit distilleries the distiller must also record the number of boilings each day, the quantity of singlings, and the quantity of brandy produced. Every distiller is required to keep a record and make monthly return, under oath, of all materials received, the quantity used each day, and the quantity of spirits produced therefrom.

Spirits to be gauged, etc.—All finished spirits produced from grain or molasses must be immediately conveyed to the receiving cisterns, and not later than the third day must be drawn into packages (unless otherwise transferred to denaturing warehouses), and, after being carefully proofed, gauged, and marked by the officer must be at once deposited in the distillery warehouse approved for that purpose.

On the first day of each month, or within five days thereafter, the distiller must file with the collector an entry, under oath, showing the quantity of spirits deposited each day during the month, and must execute a bond, with satisfactory sureties, securing the payment of tax on all spirits so deposited. This bond is in addition to the general bond which the distiller is required to file before commencing business.

Spirits produced from fruit must be immediately casked by the distiller and removed to the designated place of deposit on the distillery premises, and when inspected and properly marked by a gauger the spirits must be tax-paid or removed in bond for deposit in a special bonded warehouse, or otherwise lawfully disposed of.

Deficiency and excess.—In case the distiller fails to produce 80 per cent of the surveyed capacity of his distillery during the month, or fails to produce and account for 100 per cent as to any material used by him in excess of his survey, the tax due on such deficiency or excess is to be assessed against him. If, however, such deficiency is found to be due to unavoidable accident, or (in case of deficiency or excess) to a misunderstanding of the law and regulations, the Commissioner of Internal Revenue is authorized to remit the tax in such cases.

Industrial distilleries.—Distilleries established under the act of March 2, 1907, already referred to, are designated in the denatured alcohol regulations (Reg. No. 30, pt. 2) as industrial distilleries; and the exemptions made in favor of these distilleries (pp. 39, 40, and 41) briefly stated are as follows:

From all the provisions of—

Sec. 3263, R. S., requiring a plan of the distillery to be furnished.

Sec. 3267, R. S., respecting the number, construction, and arrangement of receiving cisterns (except as to locks) and time in which spirits are to be withdrawn from such cisterns.

Sec. 3271, R. S., requiring a distillery warehouse to be provided.

Sec. 3285, R. S., prescribing the time when fermenting tubs are to be emptied.

Sec. 3287, R. S., prescribing how spirits, when withdrawn from receiving cisterns, shall be casked and branded, and requiring such spirits, when casked, to be deposited in the distillery warehouse.

Sec. 3302, R. S., as to distilleries to which no storekeeper is regularly assigned.

Sec. 3309, R. S., providing for assessment in case of deficiency in production or excess of materials used. This provision, however, to be waived only where it satisfactorily appears that all spirits produced from the materials used have been duly accounted for.

Sec. 3310, R. S., limiting the fermenting period, except so much of said section as requires notice of suspension or resumption of work.

Also so much of—

Sec. 3259, R. S., as requires the fermenting period to be stated in the distiller's notice.

Sec. 3264, R. S., as prescribes the bases for determining, per bushel or gallon, the quantity of mash or beer at grain or molasses distilleries.

Sec. 3266, R. S., as prohibits the use of any still, boiler, or other vessel for distilling spirits in any building, yard, or inclosure connected with any dwelling house, or where any other business (except the rectification of spirits or the manufacture of fermented liquors) is carried on.

Sec. 3284, R. S., as prohibits the use of any distilling material in the absence of a storekeeper, unless a regular storekeeper has been assigned to the distillery.

Sec. 3303, R. S., as requires a record to be kept showing how materials were conveyed to premises; the amount paid for such materials; the kind and quantity of fuel used; the amount paid for repairs placed on the distillery, and the name of each person employed on the premises and the capacity in which employed.

Sec. 3311, R. S., as requires the closing and sealing of tubs, when notice of suspension of work is given.

These exemptions do not, of course, include any penalties imposed by law for defrauding or attempting to defraud the revenue in connection with the operation of distilleries.

Surveys.—As stated on page 44 of the regulations, no fixed rule can be laid down for determining the spirit-producing capacity of industrial distilleries, owing to the various kinds of stills and the variable conditions under which distilling materials may be used. As a general rule, however, the capacity of such distilleries is to be computed, as in the case of brandy distilleries, on the boiling capacity of the stills and the number of boilings that may be made in twenty-four hours. But, in order to determine whether such distilleries are entitled, under the law, to the exemptions made as to distilleries "having a daily spirit-producing capacity of not exceeding 100 proof gallons," the survey, as stated on page 42 of the regulations, will be based on the maximum quantity of spirits that can be produced from the material containing the largest per centum of spirit-yielding matter it is proposed to use.[a] In such cases, however, the distiller is to be held accountable only for the spirits actually produced by him, and not, as in the case of other distillers, for the assumed production based on the survey made. This exemption and the privilege accorded distillers of this class (pp. 53–54) to operate their distilleries in the absence of a government store-

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[a] The spirit-yielding properties of many materials that may be used in distilling spirits have not yet been determined, but computed on the per cent of fermentable matter and on the basis of 56 pounds the following-named materials should yield, approximately, the quantity of proof spirits here stated:

	Proof gallons.		Proof gallons.
Rice	5.28	Artichokes	1.15
Corn, rye, spelt, sorghum seed	4.75	Sugar beets	1.02
Barley	4.61	Sorghum and sugar cane	.81
Cassava	2.37	Turnips	.68
Sweet potatoes	1.69	Sweet corn stalks and cobs	.54
White potatoes	1.36	Cornstalks	.34

The quantity of fruit materials capable of producing 1 proof gallon of spirits is found to be, approximately—

	Gallons.		Gallons.
Apple or peach pomace	14	Sour wine, lees or piquette	7
Grape pomace	10 to 12	Wine	5
Cider	11	Wash	10 to 12.5

keeper (when found to be entitled to the confidence of the Department, and without reference to the kind of materials to be used) remove many of the seemingly onerous requirements and restrictions which have been found necessary, as to ordinary distilleries, to protect the revenue against fraud. Such industrial distilleries, however, will be under the supervision of a general storekeeper-gauger.

Owing to the high rate of tax imposed on distilled spirits, the temptation to evade this tax is very great, and while all governmental regulations are more or less burdensome to the citizen, it is obviously necessary for the Government to adopt such safeguards, especially in the matter of distilling spirits, as will prevent, so far as possible, unscrupulous persons from defrauding the revenue.

The requirements of the regulations relating to industrial distilleries, briefly stated, are—

(1) The registry of each still to be used.

(2) The erection of an alcohol room and cisterns for the storage of the alcohol produced.

(3) The filing of the prescribed notice and the giving of the required bond.

(4) The keeping of records showing the materials received on the distillery premises and the quantity used; the quantity of spirits produced therefrom and the quantity disposed of; and the furnishing of a sworn transcript of such records each month.

The regulations also require that the distiller shall be the owner of the premises used, or file consent of the owner; that all stills, tubs, and cisterns shall be numbered and provided with locks; that all pipes shall be painted; and that a material room and mixing tank shall be provided when necessary. Sections 20 and 22 (p. 48), however, provide for the storage of denaturants in the alcohol room and for the denaturation of alcohol in ordinary casks or packages in certain cases. The regulations further provide (p. 58) the manner in which approved denaturants may be procured and used.

Agricultural experiment distilleries.—Under supplemental regulations (Treasury Department Circular 29 of April 27, 1908) stills for the manufacture of alcohol for denaturation only may be established and operated at any agricultural experiment station, established under the laws of any State or Territory. Such distilleries, designated as "agricultural experiment distilleries," must be registered and must be operated under the supervision of a regularly designated officer of the United States Department of Agriculture or the experiment station. Such officer is required to file the prescribed notice and give a satisfactory bond; and the alcohol when produced, unless conveyed to a locked storage tank, must be at once completely denatured. When denatured, the alcohol may be used on the premises for fuel, light, or power, or may be sold or otherwise disposed of in packages to be duly marked and stamped by an officer to be detailed for that purpose by the collector of the district. These regulations also require a monthly statement to be rendered, showing the kind and quantity of materials used, the fermenting period, the number of boilings each day, the quantity of alcohol produced, the kind and quantity of denaturants used, and the disposition of the alcohol denatured.

Sawdust and cactus distilleries.—So far no industrial distilleries have been registered. In May, 1906, or just prior to the enactment of the Denatured Alcohol Law of June 7, 1906, a distillery was established for the manufacture of spirits from sawdust, and, while surveyed on the basis of 6 per cent only, deficiencies were incurred, the average yield being considerably less than 80 per cent of the survey. Operations at this distillery were suspended in September, 1906. Recently a distillery, producing spirits from the cactus plant, has been established. This distillery is also surveyed at 6 per cent, but the returns showing the average yield so far obtained are not now available.

Such distilleries having, under the survey made, a daily spirit-producing capacity exceeding 100 proof gallons, can not, under the act of March 2, 1907, be registered and operated as industrial distilleries.

CLOSING REMARKS ON THE DENATURED ALCOHOL COURSE.

By H. W. WILEY, *Bureau of Chemistry*.

In closing the course of lectures on denatured alcohol, I wish to express our appreciation of the contributions made by the gentlemen not attached to this Bureau, but in other branches of the public service, and to those not in the public service at all, who have taken the trouble to come here and give us the results of their knowledge and experience on this subject. The course has presented not only the technical and theoretical conditions which arise in the manufacture of alcohol, but also the practical exposition of its manufacture by those who are engaged in the industry, so that we have both the theory and the practice combined. In order to illustrate more pointedly the character of this alcohol, we have clearly differentiated between alcohol used for industrial purposes and alcohol used for beverages as they are produced in this and other countries. The question has been presented thus in all of its aspects. In addition to this, specialists have lectured on the application of denatured alcohol for different purposes; especially for power and for use in the arts, as illustrated in the lectures of Mr. Strong, Mr. Veitch, and Doctor Monroe.[a]

It may seem from what has been said that there is no treatment to which an alcoholic liquor can be subjected which will prevent its being drunk by some degenerate persons. The problem of denaturing is not to put a deadly poison into alcohol, but to give it such an unpleasant odor and taste that any normal person would not use it as a beverage. Just as there are many persons who would, if not restrained in some way, use pure alcohol as a beverage, so some would drink industrial alcohol unless it were made objectionable in some way. And it is to this end that the denaturants that have been mentioned are added. You have heard how the Commissioner of Internal Revenue has endeavored to meet all the requirements of the trade by permitting the use of a denaturant in each particular case which would not interfere with the technical use of the product. Our law, as you have been told by the commissioner and by Doctor Crampton, is more liberal in that respect than the laws of other countries.

I believe that it has been made perfectly clear, particularly by the observations of Doctor Crampton and Commissioner Capers, that the very small still making 10 and 15 gallons of proof alcohol per day is not one which can be looked forward to as a means of solving the problem of manufacture on a small scale. The still which is to be successful will be the one with not less than 100 gallons per day capacity and probably more, which can be utilized by the neighborhood for the purpose of working up the wastes of the farm. Hundreds of carloads of apples rot in Missouri and Kansas simply because it will not pay to transport them, at the prices received, to distant markets. In Georgia, hundreds of carloads of peaches which are not suitable to send to distant markets are allowed to go to waste, and watermelons are left to spoil on the ground. Our experience here, however, shows that it takes a good deal of experimental work to determine just how these fruits may be profitably worked into alcohol, but it is not an impossible problem. There is no difficulty in devising means whereby all of the fermentable matter of such wastes and by-products may be brought into use, therefore incidentally and for the farmers' own use they may be of great value. But I believe it has been shown that on a large scale, for actual profit, and for manufacturing alcohol in commercial quantities, the farmers of this country must grow particularly the things that are used for this purpose. The sweet potato, the yam, cassava, and damaged grain in all parts of this country, and the refuse of sugar factories, perhaps most of all, are to be looked to as sources of denatured alcohol, at the

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[a] The latter lecture, unfortunately, was not adapted to reproduction, owing to its intimate dependence on the exhibits shown.

price which will place the manufacturer on even terms with those who use coal and petroleum products.

The expectations which were held at the time of the passage of this act have not been realized; the hope of the future lies not only in the utilization of the waste which is most convenient but in the production of a crop particularly for this purpose. There are quantities of sugar beets in this country which have only a low percentage of sugar, and yet can be very successfully employed, as in France, in the manufacture of industrial alcohol. The potato should be grown in large quantities, and in this way our farmers could secure a cheap and profitable source of alcohol. The statistics show (see p. 97) that the average yield of the potato in this country is less than 100 bushels per acre, while in Germany it is two and a half times that amount. Our farmer must learn to grow 300 bushels to the acre instead of 100. Five hundred bushels have been grown on a poor Maryland sand field, which shows what an intelligent farmer can do even on poor soil. The prospects for the growth of the sweet potato and the yam in the sand lands of the Southern States are flattering, and though those lands are poor in one sense, they are yet very productive with a small amount of fertilizing. Mr. Moore called attention to the crops of cassava which grow very luxuriantly in sandy soils, and there are also the watermelon and peach crops, and the molasses output of the South, which may become very productive in this industry. The Central States grow the Irish potato, which will yield from 20 to 21 per cent of fermentable matter. Some potatoes are not well suited for table use but give large quantities of fermentable material. With all of these resources there seems to be no reason why the agricultural distillery should not flourish in this country as does the farm distillery in Germany and France. In Great Britain, on the other hand, there are no small stills of any description where alcohol of any kind is made. They depend upon the large distilleries for their alcohol and import both raw materials and the alcohol itself, German spirits, so-called, being very plentiful in Great Britain, while a large part of the silent spirits or grain whisky is made from Indian corn imported from the United States. The English conditions are not propitious for the institution of the agricultural still, but our country presents some of the same advantages as Germany and France for the development of this industry.

We have exhibited here an experimental still making about 100 gallons of proof spirits a day, but this is not the type of still, it seems to me, that the agricultural still must be. If the production of alcohol of a lower strength should be permitted, an even simpler still might be used. I was much interested to hear Mr. Strong say that alcohol of 80 per cent proof and even lower could be used for heat and light. That would make it possible to use a pot still, which is the simplest and least expensive form of all, and would thus aid in solving the problem of distillation, as the production of alcohol above 80 per cent is an expensive process.

If only a few of the States will take up the production of industrial alcohol, others will soon follow, and a general interest in the problem will be disseminated throughout the country. It was in this hope that this course was organized, these lectures arranged, and the technical demonstrations made. In this way a substantial beginning may be made in this country in the manufacture of denatured alcohol, thus securing the full benefit which was intended in the enactment of the law.

APPENDIX.

INTERNAL-REVENUE CIRCULAR NO. 721, RELATING TO THE ESTABLISHMENT AND OPERATION OF STILLS AT AGRICULTURAL EXPERIMENT STATIONS FOR THE MANUFACTURE OF DENATURED ALCOHOL.

Section 4 of the act of March 2, 1907, relating to denatured alcohol provides:

"That at distilleries producing alcohol from any substance whatever, for denaturation only, and having a daily spirit-producing capacity of not exceeding one hundred proof gallons, the use of cisterns or tanks of such size and construction as may be deemed expedient may be permitted in lieu of distillery bonded warehouses, and the production, storage, the manner and process of denaturing on the distillery premises the alcohol produced, and transportation of such alcohol, and the operations of such distilleries shall be upon the execution of such bonds and under such regulations as the Commissioner of Internal Revenue, with the approval of the Secretary of the Treasury, may prescribe, and such distilleries may by such regulations be exempted from such provisions of the existing laws relating to distilleries as may be deemed expedient by said officials."

Under and by virtue of the authority conferred by said section 4, distilleries having a daily spirit-producing capacity of not exceeding 100 proof gallons,[a] established by the Department of Agriculture at any of its experiment stations, or by any agricultural college of any State or Territory at any experiment station established under the laws of such State or Territory, for the production of alcohol for denaturation only, are hereby exempt from any and all provisions of existing law relating to distilleries, except the provisions of sections 3258 and 3264, Revised Statutes, relating to the registry of stills and the survey of distilleries, and so much of section 3259 as requires every person intending to engage in the business of a distiller to give notice in writing to the collector of the district where the business is to be carried on. Distilleries so established will constitute a separate and distinct class, to be designated as "Agricultural Experiment Distilleries," and will be subject to the following regulations:

1. Each such distillery shall be operated under the supervision of a regularly designated officer of the Department or college, who shall file with the collector of internal revenue of the district a registration in duplicate, on Form 26, of the stills when set up for use. Such officer shall also file with the collector a notice in duplicate setting forth—

(a) Name and location of the Department, or college, establishing such distillery.
(b) The exact location of the distillery.
(c) The kind of each still and its cubic contents in gallons.

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[a] The daily spirit-producing capacity of such distilleries will be determined on the basis of twenty-four hours, as fixed by section 3264, Revised Statutes, and the lowest spirit-producing material to be used in operating such distilleries.

(*d*) The kind of material to be used; the estimated fermenting period for each kind of material, and the estimated quantity of alcohol, in proof gallons, to be produced each day from each.

(*e*) The formula under which the alcohol so produced is to be denatured, and—

(*f*) The size, description, and location of the building or tank in which the alcohol produced is to be denatured and stored.

2. On filing the foregoing notice such officer will also file with the collector a bond in duplicate in a penal sum of not less than $1,000 and with sureties satisfactory to the collector. Such bond will be in the following form:

Know all men by these presents, That we, ——, of ——, as principal, and ——, of ——, and ——, of ——, as sureties, are held and firmly bound unto the United States of America in the full and just sum of —— thousand dollars, for the payment whereof to the United States we bind ourselves, our several heirs, executors, administrators, and assigns, jointly and severally, firmly by these presents.

Witness our hands and seals at ——, on this —— day of ——, 190—.

Whereas the above bounden principal, for and on behalf of the ——, has, under the provisions of section 4 of act of Congress approved March 2, 1907, and regulations issued pursuant to said act, registered with the collector of internal revenue of the —— district of ——, a distillery located at ——, county of ——, State of ——, in said district, and whereas the said principal intends for and on behalf of the said —— to operate said distillery for the purpose of conducting experiments in the manufacture of alcohol for denaturation only.

Now, therefore, the condition of this obligation is such that if all the requirements of said law and regulations respecting the operations carried on at said distillery, including the production, denaturation, storage, and use of all alcohol or spirits on said distillery premises, are fully complied with, and if the said distillery while so registered shall be used for no purpose other than that authorized by said act and regulations, then this obligation to be void; otherwise to remain in full force and virtue.

——— ——— (SEAL).
——— ——— (SEAL).
——— ——— (SEAL).

Signed, sealed, and delivered in the presence of—

——— ———

——— ———

Upon the approval of the bond by the collector the duplicate thereof, together with the duplicate notice filed, will be forwarded to the Commissioner of Internal Revenue, and the distillery so registered and bonded may thereafter be operated for the purpose described in the notice, subject to the following requirements and limitations:

3. The alcohol produced must be not less than 180° proof; and, unless conveyed directly to locked storage tanks, must be at once completely denatured (i. e., during the same day when produced) under formula 1 or 2, prescribed on page 23 of Regulations 30, revised July 15, 1907.

If conveyed to locked storage tanks, the alcohol must be likewise denatured and not later than the last day of the month during which it is produced.

When so denatured, the alcohol may be used on the distillery premises for fuel, light, or power, or for any experimental purpose, or may be sold or otherwise disposed of after the same has been denatured and the packages containing the same have been duly marked and stamped by an officer to be detailed for this purpose by the collector of the district. Under no circumstances should the alcohol when denatured be recovered by redistillation or by any other process, as such recovery (except in the case of manufacturers, where the alcohol is to be reused for manufacturing purposes) is expressly prohibited by section 2, act of June 7, 1906.

4. At the end of each month, or within ten days thereafter, the officer under whose supervision the distillery was operated during the month will furnish the collector

of the district a written statement showing (a) the kind and quantity of materials u.ed during the month, (b) the fermenting period of each kind of material used, (c) the number of boilings each day the distillery was operated, and the quantity of alcohol, in wine and proof gallons, produced each day from each kind of material used, (d) the kind and quantity of denaturants used and the quantity of alcohol (in wine and proof gallons) denatured during the month, and (e) the disposition of the alcohol so denatured.

The business so carried on at such distilleries will at all times be subject to inspection by the internal-revenue officers.

The foregoing regulations will take effect May 1, 1908, and until printed blanks of the forms herein prescribed are furnished, typewritten forms may be used.

WASHINGTON, D. C., *April 27, 1908.*

SPECIAL DENATURANTS.[a]

[In addition to the general denaturants adopted (p. 63) the following seventeen formulas for special denaturants had been authorized up to November 24, 1908, for use in certain industries, also enumerated, which could not use the general formulas without deleterious effect upon the product.]

ART. 25. The Commissioner of Internal Revenue will consider any formula for special denaturation that may be submitted by any manufacturer in any art or industry, and will determine (1) whether or not the manufacture in which it is proposed to use the alcohol belongs to a class in which tax-free alcohol withdrawn under the provisions of the law can be used; (2) whether or not it is practicable to permit the use of the proposed denaturant and at the same time properly safeguard the revenue. But one special denaturant will be authorized for the same class of industries, unless it shall be shown that there is good reason for additional special denaturants.

The commissioner will announce from time to time the formulas of denaturants that will be permitted in the several classes of industries in which tax-free alcohol can be used.

Up to date the following formulas have been authorized, to wit:

To 100 gallons of ethyl alcohol add—

No. 1.—Five gallons of approved wood alcohol, the wood alcohol to be subject to the same specifications as are imposed upon the methyl alcohol used in completely denatured alcohol, which are set out in section 26, Part I, Regulations No. 30.

No. 2.—Seven pounds of camphor and 5 gallons of commercially pure methyl alcohol, the methyl alcohol to have a specific gravity of not more than 0.810 at 60° F.

No. 2a (alternative).—Two gallons of approved wood alcohol and 2 gallons of benzol.

The wood alcohol shall be subject to the same specifications as are imposed upon the wood alcohol used in completely denatured alcohol, which are set forth in section 26, Part I.

The benzol shall be subject to the following specifications:

Solubility in water.—When 10 cc. of benzol are shaken with an equal volume of water in a glass-stoppered cylinder, divided into tenths of a cubic centimeter, and allowed to stand five minutes to separate, the upper layer of liquid must measure not less than 9.5 cc.

Boiling point.—When 100 cc. of benzol are subjected to distillation in the same manner as described for the determination of the boiling point of wood alcohol in sec-

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[a] From Regulations No. 30, Supplement No. 1, United States Internal Revenue.

tion 26, Part I, not more than 1 cc. should go over at 77° C., and not less than 90 cc. at 100° C.

No. 3.—Six and one-half gallons of the following mixture: Five gallons of commercially pure methyl alcohol, having a specific gravity of not more than 0.810 at 60° F.; 1 gallon of castor oil; one-half gallon of 36° Baumé caustic soda lye.

The denaturing mixture is best prepared by dissolving the castor oil in the methyl alcohol, and then adding the soda lye.

No. 4.—One gallon of the following solution: Ten gallons of an aqueous solution containing 40 per cent nicotine; 0.4 pound acid, yellow dye (fast yellow Y); 0.4 pound tetrazo brilliant blue, 12 B. Conct.; water to make 100 gallons.

The tobacco denaturant must conform to the following analytical requirements:

Determination of nicotine.—It must contain not less than 3.75 per cent of nicotine when tested by the following process:

Ten cc. of the solution are measured into a 500 cc. Kjeldahl flask, provided with a suitable bulb tube, 10 cc. of N/10 alkali added, the liquid made up to 50 cc., and distilled in a current of steam until the distillate is no longer alkaline (about 500 cc.). The distillate is then titrated with N/10 sulphuric acid, using rosolic acid as an indicator. Not less than 23.2 cc. should be required for the neutralization.

Test of coloring matter.—Take 1 cc. of the denaturant and make up to 100 cc. with water, acidulating with a few drops of sulphuric acid. Immerse in this solution a piece of white cotton cloth and boil the solution. Continue the process, adding more cloth and more water if necessary, until all the blue color in the solution is fixed on the cloth. Then add a piece of white woolen cloth, and boil the bath as before, until all the yellow color is fixed upon the cloth. Both the cotton and woolen cloths should show decided colors—the cotton blue and the woolen yellow.

Intensity of color.—The denaturant solution, when observed in an eighth-inch cell of Lovibond's tintometer, must show a color of an intensity not less than No. 24 yellow, combined with No. 3 blue.

No. 5.—Sixty-five pounds of sulphuric ether, 3 pounds of cadmium iodid, and 3 pounds of ammonium iodid.

No. 6.—Three gallons commercially pure methyl alcohol and one-half gallon pyridin bases; the methyl alcohol to have a specific gravity of not more than 0.810 at 60° F.; the pyridin bases to conform to specifications as set forth in section 26, Part I, Regulations No. 30.

No. 6a (alternative).—Fifteen gallons of condensed fumes, recovered in the process of manufacture. The condensed liquid shall be subject to the following specifications:

It must contain not less than 4 grams per 100 cc. of mixed aldehydes calculated as acetic aldehyde.

The use of this alternative formula will be restricted to factories operating in connection with either a distillery or a central denaturing bonded warehouse.

No. 7.—Revoked. (Formula No. 1 substituted.)

No. 8.—One gallon of pyridin bases and 1 gallon of benzol. The pyridin bases to conform to specifications set forth in section 26, Part I. The benzol to be subject to the specifications contained in formula No. 2a.

No. 9.—Ten gallons of acetone and 2 gallons of petroleum naphtha. The petroleum naphtha to have a specific gravity of not less than 0.650 nor more than 0.720 at 60° F.

No. 10.—Two gallons of approved wood alcohol and 2 gallons of benzol. The wood alcohol shall be subject to the same specifications as are imposed upon the methyl alcohol used in completely denatured alcohol, which are set forth in section 26, Part I. The benzol to conform to the specifications imposed in formula No. 2a.

No. 11.—One hundred pounds of sulphuric ether and 10 pounds of cadmium iodid.

No. 12.—One gallon of pyridin bases and 2 gallons of coal-tar benzol.

The pyridin bases must conform to the specifications set forth in section 26, Part I, and the benzol must conform to the specifications contained in formula No. 2a.

No. 13.—Five gallons of sulphuric acid having a specific gravity of not less than 1.83 at 60° F., and 5 gallons of sulphuric ether having a specific gravity of not more than 0.728 at 60° F.

No. 13a (alternative).—Ten gallons of sulphuric ether, having a specific gravity of not more than 0.728 at 60° F.

This alternative denaturant is authorized because of representations made by manufacturers to the effect that formula No. 13 would give rise to many difficulties in the production of the anesthetic grade of ether. It is not as efficient a denaturing material, however, and internal-revenue officers are hereby instructed to exercise great caution in recommending the granting of permits for its use, both as to the standing and the responsibility of the applicant and the character of the premises described in the application. A very careful and close surveillance of such factories must be maintained.

The use of this alternative formula is further restricted to the manufacture of sulphuric ether containing not more than 4 per cent of alcohol by weight, and section 40, Part VI, of the revised regulations, is amended accordingly.

No. 14 (alternative).—Five gallons commercially pure methyl alcohol and 10 pounds anhydrous zinc chlorid.

The methyl alcohol must have a specific gravity of not more than 0.810 at 60° F.

No. 15.—Three gallons of sulphuric acid having a specific gravity of not less than 1.83 at 60° F., and 1 gallon of kerosene having a specific gravity of not less than 0.800 at 60° F.

No. 16.—Five gallons commercially pure methyl alcohol and 2 gallons benzol.

The methyl alcohol shall have a specific gravity of not more than 0.810 at 60° F.

The benzol shall be subject to the same specifications as are imposed in formula No. 2a.

Formula No. 17.a—To 100 gallons of ethyl alcohol add 0.05 gallon (6½ fluid ounces) of animal oil.

The animal oil must conform to the following specifications:

Color.—The color shall be a deep brown.

Boiling point.—When 100 cc. of the animal oil are subjected to distillation in the same manner as prescribed for the determination of the boiling point of wood alcohol in section 26, Part I, of the regulations, not more than 5 cc. should distil over below 90° C., and not less than 50 cc. at 180° C.

Pyrrol reaction.—Two and one-half cubic centimeters of a 1 per cent solution of the animal oil in 90 per cent of alcohol by volume are diluted to 100 cc. with alcohol. A splinter of pine wood, previously moistened with concentrated hydrochloric acid, is dipped into 10 cc. of this solution, containing 0.025 per cent of animal oil. After a few minutes the splinter should show a distinct red coloration.

Reaction with mercuric chlorid.—Five cubic centimeters of the 1 per cent solution of the animal oil in 90 per cent of alcohol by volume, when treated with 5 cc. of a 2 per cent solution of mercuric chlorid in alcohol, should give immediately a voluminous flocculent precipitate. Five cubic centimeters of the 0.025 solution of the animal oil, when treated with 5 cc. of the 2 per cent solution of mercuric chlorid, should show at once a distinct turbidity.

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a T. D. 1439, November 24, 1908.

These formulas have been authorized for use in the various industries as indicated in the following table:

Manufacture or preparation of—	Formula No.
Alkaloids and fine chemicals	16
Acetic ether	1

(Provided that the finished product contains not more than 6 per cent of alcohol by volume.)

Barometer and thermometer tubes	1
Celluloid, pyralin, and similar products	2–2a
Chloral hydrate	17
Confectioners' colors	1
Embalming fluids	1
Ethyl chlorid	1, 14
Filaments for incandescent electric lamps	1
Fine chemicals and alkaloids	16
Fulminate of mercury	6–6a
Heliotropin	1
Imitation leather	12
Jewelry and watches	1
Lacquers, pastes, and varnishes from soluble cotton	1, 10
Moldings and picture frames	1
Monobromated camphor	9
Nitrous ether	15

(After the tax-free alcohol has all been converted into nitrous ether it will be permissible for the manufacturer to add alcohol in order to dilute the product so that it may be more readily transported, or for other purposes, but such added alcohol must in all cases be tax-paid alcohol.)

Pyralin, celluloid, and similar products	2–2a
Pastes, lacquers, and varnishes from soluble cotton	1, 10
Photographic collodion	11, 15
Photographic dry plates	1
Photo-enlargements and photo-prints	5
Photo-engravings	5
Postal cards in colors	1
Resin of podophyllum and similar products	1
Rubber, purification of	9
Santonine	9
Shellac varnishes	1
Shoe polish	1
Silverware and bronze	1
Soap, transparent	1, 3
Sulphonmethane	8
Strychnine	9
Solid and powdered medicinal extracts	1
Sulphuric ether	13–13a
Surgical ligatures	1
Tannic acid	9
Thermometer and barometer tubes	1
Tobacco, smoking and chewing	4
Transparent soap	1, 3
Varnishes, pastes, and lacquers from soluble cotton	1, 10
Watches	1

STATISTICS OF DENATURED ALCOHOL PRODUCTION, FROM THE ANNUAL REPORT OF THE COMMISSIONER OF INTERNAL REVENUE FOR THE FISCAL YEAR ENDED JUNE 30, 1908.

Statement showing the quantity (in wine gallons) of denatured alcohol produced at and shipped from denaturing bonded warehouses, also alcohol redenatured at restoring and redenaturing plants, during the fiscal year ended June 30, 1908.

District.	Number in district.	Produced. Completely denatured.	Produced. Specially denatured.	Shipped to manufacturers. Completely denatured.	Shipped to manufacturers. Specially denatured.
First California	1	109,704.83	17,713.92	5,947.22	17,713.92
Fifth Illinois a	4	1,415,638.37	b 1,087,607.25	5,376.58	968,189.16
Sixth Indiana	1	66,655.80	66,579.01	23,423.87	66,579.01
Seventh Indiana c	1	70,981.44	40,358.24	21,056.54	26,728.36
Sixth Kentucky	1	....	44,454.00	....	44,454.00
Louisiana d	1	91,005.43	40,044.95	....	....
Third Massachusetts	2	e 56,519.28	f 211,478.05	1,169.24	206,159.31
First Wisconsin	1	1,617.23	1,093.93	791.78	1,093.93
Total	12	1,812,122.38	1,509,329.35	57,765.23	1,330,917.69
Statement for six months ended June 30, 1907	8	1,397,861.16	382,415.19	181,759.28	375,276.94

District.	Denaturing bonded warehouses. Shipped to dealers and others. Completely denatured. Wholesale dealers.	Retail dealers.	Others.	Specially denatured. Wholesale dealers.	Restoring and redenaturing plants. Number in district.	Restored and completely denatured.	Restored and specially denatured.
First California	103,757.61	....	....	....	......	....	....
Connecticut	....	....	....	....	1	33,469.71	84,118.90
Fifth Illinois a	1,377,115.20	12,903.93	20,242.66	115,686.69	......	....	....
Sixth Indiana	34,430.12	5,211.47	3,590.34	....	......	....	....
Seventh Indiana c	42,244.22	1,028.25	6,652.43	13,629.88	......	....	....
Louisiana d	91,005.43	....	....	40,044.95	......	....	....
Third Massachusetts	53,970.12	724.16	....	1,077.54	......	....	....
First Wisconsin	717.19	34.62	73.64	....	......	....	....
Total	1,703,239.89	19,902.43	30,559.07	170,439.06	......	33,469.71	84,118.90
Statement for six months ended June 30, 1907	1,149,029.24	7,057.83	17,916.84	....	1	27,483.19	....

a One discontinued April 30, 1908.
b On hand in casks 3,731.40 wine gallons specially denatured alcohol.
c Discontinued March 31, 1908.
d Central denaturing bonded warehouse.
e On hand in casks 655.76 wine gallons completely denatured alcohol.
f On hand in casks 4,241.20 wine gallons specially denatured alcohol.

Statement showing quantity of specially denatured alcohol used for industrial purposes under different formulas during the fiscal year ended June 30, 1908.

[Quantities in wine gallons.]

	Article manufactured.	Quantity.
Formula No. 1: 100 gallons of ethyl alcohol and 5 gallons approved wood alcohol.	Alkaloids and fine chemicals; acetic ether barometer and thermometer tubes, confectioners' colors; embalming fluid; ethyl chloride; filaments for incandescent lamps; heliotropin; inks; jewelry and watches; lacquers, pastes, and varnishes from soluble cotton; moldings and picture frames; photographic dry plates; postal cards in colors; resin of podophyllum and similar products; santonine; shellac varnishes; shoe polish; silverware and bronze; transparent soap; soldering flux; strychnine; solid and powdered medicinal extracts; surgical ligatures.	768,476.63
Formula No. 2: 100 gallons of ethyl alcohol, 7 pounds camphor, and 5 gallons of commercially pure methyl alcohol.	Pyralin and similar products..........	60,144.07
Formula No. 2a (alternative): 100 gallons of ethyl alcohol, 2 gallons of approved wood alcohol, and 2 gallons of benzol.	.....do................................	35,097.24
Formula No. 3: 100 gallons of ethyl alcohol, to which is added 6½ gallons of the following mixture: 5 gallons commercially pure methyl alcohol, 1 gallon castor oil, one-half gallon 36° Baumé caustic soda lye.	Transparent soap................:......	63,520.68
Formula No. 4: 100 gallons of ethyl alcohol, to which is added 1 gallon of the following solution: 10 gallons of an aqueous solution containing 40 per cent nicotine, 0.4 pound acid, yellow dye (fast color Y), 0.4 pound tetrazo brilliant blue, 12 B Conct., water to make 100 gallons.	Tobacco, smoking and chewing........	288,981.36
Formula No. 5: 100 gallons of ethyl alcohol, 65 pounds sulphuric ether, 3 pounds cadmium iodide, and 3 pounds ammonium iodide.	Photo enlargements and photo prints; photo-engravings; photographic collodion.	524.11
Formula No. 6: 100 gallons of ethyl alcohol, 3 gallons commercially pure methyl alcohol, and one-half gallon pyridin bases.	Fulminate of mercury.................	31,946.49
Formula No. 6a (alternative): 100 gallons of ethyl alcohol and 15 gallons of condensed fumes recovered in process of manufacture.	.....do................................	None used.
Formula No. 7 (revoked and formula No. 1 substituted).	Watches...............................	186.39
Formula No. 8: 100 gallons of ethyl alcohol, 1 gallon pyridin bases, 1 gallon benzol.	Sulphonmethane......................	190.19
Formula No. 9: 100 gallons ethyl alcohol, 10 gallons acetone, and 2 gallons petroleum naphtha.	Monobromated camphor; rubber (purification of); santonine; strychnine; tannic acid.	3,930.48
Formula No. 10: 100 gallons ethyl alcohol, 2 gallons approved wood alcohol, and 2 gallons benzol.	Lacquers, pastes, and varnishes from soluble cotton.	None used; No. 1 substituted.
Formula No. 11: 100 gallons ethyl alcohol, 100 pounds sulphuric ether, and 10 pounds cadmium iodide.	Photographic collodion...............	332.95
Formula No. 12: 100 gallons ethyl alcohol, 1 gallon pyridin bases, 2 gallons coal-tar benzol.	Imitation leather.....................	34,129.43
Formula No. 13: 100 gallons ethyl alcohol, 5 gallons sulphuric acid, and 5 gallons sulphuric ether.	Sulphuric ether.......................	None used; No.13a substituted.
Formula No. 13a (alternative): 100 gallons ethyl alcohol, 10 gallons sulphuric ether.	.....do................................	211,495.49
Formula No. 14: 100 gallons ethyl alcohol, 5 gallons commercially pure methyl alcohol, and 10 pounds anhydrous zinc chloride.	Ethyl chloride........................	264.16
Formula No. 15: 100 gallons ethyl alcohol, 3 gallons sulphuric acid, and 1 gallon kerosene.	Nitrous ether.........................	1,582.34
Formula No. 16: 100 gallons ethyl alcohol, 5 gallons commercially pure methyl alcohol, and 2 gallons benzol.	Alkaloids and fine chemicals..........	554.44
	Total...............................	a 1,501,356.45

a Not including 7,972.60 wine gallons in casks at denaturing warehouses at the close of the year.